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Bachelor of Engineering Thesis

Microstructure and Mechanical properties of titanium alloys in biomedical application

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Abstract

One of the largest issues facing the biomedical industry today is the rejection of titanium alloy implants within the human body, which has been known to cause severe problems in both the short- and long-term. The issue stems from a multitude of reasons, mainly being a great difference in elastic modulus between the titanium alloy and the adjacent bone that the implant is attached to. The objective of the thesis would then be to research and develop a method that would lower this gap in elastic modulus.

Through extensive research, it was found that beta-stabilisation was the more common method in reducing the elastic modulus, but other factors also had to be considered such as corrosion- and wear-resistance. A number of metals were then researched and judged based on a criterion, with the winning candidate being molybdenum for its ability to lower elastic modulus and increase corrosion- and wear-resistance.

Three compositions of titanium alloys were then developed through powder metallurgy and cold sintering, resulting in Ti-5Mo, Ti-10Mo and Ti-15Mo. From the sample compositions, four batches of samples would undergo different forms of testing: two for compression testing, one for hardness testing and one for microstructural analysis.

Microstructural analysis proved that as molybdenum content increased, beta-stabilisation in the titanium alloy also increased. However, when relating this to the results of compression testing, it was found that Ti-5Mo had the lowest elastic modulus, which was found to have alpha plus beta phase. This led to the conclusion that beta-stabilisation to lower elastic modulus did not necessarily define as beta-dominance, but rather a mere presence in the titanium alloy. Molybdenum was also found to have increased the surface hardness, resulting in greater wear-resistance. Corrosion-resistance could not be tested, but was thus believed to have increased due to the protective-film that molybdenum imparts to titanium’s surface.

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1. Introduction

The introduction of this report will cover the background of implants and how they developed over history, before leading onto the aim of the thesis.

1.1 Background

The background section will cover the history, present day and definition of biocompatibility.

1.1.1 History

Early forms of implants occurred as far back as 3,000 B.C., where the Egyptians used small pieces of wood to replace missing teeth. There are even records of coconut shells being used for minor skull injuries that took place during the building of the pyramids [1].

By 1891, the first successful hip replacement took place in Germany, with the use of ivory as the implant material. It was noted to react well with the human tissue due to ivory being a natural material that develops from mammals such as elephants and rhinoceroses [1].

It was only during the Second World War, that doctors had realised the true importance of biological acceptance into a body. This occurred during an operation on a fighter pilot who had crashed his plane, and was rescued by field medics. Upon inspection of the notable injuries, the surgeons had noticed that Perspex from the plane’s cockpit had been partially absorbed into the pilot’s eye. This became a milestone from the perspective that synthetic and modified materials could in fact be made to be compatible with the human body [1].

Despite the aforementioned historical milestones, the most relevant and important to the current thesis took place during the 1950’s, when a Swedish doctor named Per-Ingvar Brånemark made an unexpected discovery.

During one of his experiments, Brånemark used an optical device encased in titanium to survey the flow of blood within a rabbit. After collecting the required data over a period of time, Brånemark suddenly found difficulty removing the device from the rabbit’s leg. Not only had the titanium casing been accepted by the body through lack of reaction, but it was also being absorbed into the bone structure through a form of cellular merging [2].

Soon after, several of Brånemark’s students volunteered to have similar titanium-encased devices attached to their arm or leg bones, with none of them showing any signs of rejection to the implant even after a period of 6 months [2]. He would later come to term the integration of titanium into bone tissue as osseointegration.

Despite the results of his testing, many in the medical industry believed Brånemark’s work to be too unconventional, as the common belief at the time was that non-biological materials would eventually cause inflammation and inevitable rejection within the body [2]. It was because of this that Brånemark found it difficult to find funding in Sweden.

It wasn't until early 1960 that the US National Institute of Health stepped in to fund Brånemark's work, allowing him to even work with a team of scientist from a multitude of fields ranging from physicians and dentists, to engineers and metallurgist.

Finally, by 1965, a Swedish man named Gösta Larsson became Brånemark's first dental patient to receive a titanium implant. Larsson was born with a deformed jaw that made it difficult to have new teeth inserted, as well as causing difficulty to eat and talk. The titanium implant developed by Brånemark and his team allowed for better teeth insertion and alignment, which in turn allowed for Larsson to talk and eat normally for the first time in his life [2].

In 2006 when Larsson had passed away, doctors found that the titanium implant was still in good condition even after 41 years. It was only then that the medical industry realised that Brånemark had not only proved the belief at the time was incorrect, but laid the foundations for a completely new sector in the medical world.

1.1.2 Present Day Issues

Titanium alloys are now one of the most commonly used metals in modern day medical application, primarily used as orthopaedic implants due to its ability to handle high load bearing and high corrosion-resistance. Among the first of the titanium alloys to be developed were Ti-6Al-4V (Ti64) and Ti-Cr, which also happened to be the first of titanium alloys to cause major issues [3].

Ti64 was a titanium alloy originally developed for aerospace application, providing high durability, strength, high elastic modulus and corrosion resistance [3]. Although many of the aforementioned mechanical properties are ideal for implants, it was elastic modulus that presented the first problem for long-term use.

The human bone has an elastic modulus, or more commonly known as stiffness, within the range of 4-30 GPa, while Ti64 has an elastic around 125 GPa. Such a great difference in stiffness can eventually cause problems such as osteomalacia, a condition that causes bone to soften and deteriorate, often due to a severe deficiency in vitamin D [3]. Ti64 causes this from constant contact of a stiffer material with bone, as the brittleness of the implant on the bone eventually causes the bone's surface to wear away over time [3].

Another issue that arises from high difference in elastic modulus is the 'stress-shielding effect'. Stress-shielding is similar to osteomalacia, where both are the result of a stiffer material rubbing against human bone. However, in the case of stress shielding, dead bone cells appear around the bones adjacent to the implant, and cause a reaction in the body which encapsulates the implant in a soft tissue to protect the body [4]. This prevents the implant from being bonding and mechanically interlocking with the adjacent bone, eventually leading implant loosening [4].

Unfortunately, high difference in elastic modulus isn't the only complication that is caused by present day titanium alloys. Ti64's aluminium content allows it to produce an oxide film with the oxide film produced by titanium to make an extra strong corrosion-resistant layer[3]. Despite this being an ideal feature, the oxide ions produced from aluminium have been known to be very reactive in the body, causing minor chemical reactions with the blood that flows to the brain. Over a long period of time, this reaction in the blood eventually causes conditions such as Alzheimer's disease [3].

It was only when a large number of cases where the aforementioned problems arose, that medical experts began to focus on methods to better increase the compatibility between implant and body. Thus, the study of increasing biocompatibility in modern titanium-alloy implants was created.

1.1.3 Biocompatibility

Biocompatibility is the measure of how well the body accepts a foreign object without rejection, and how well the object reacts in the body without causing side-effects [3]. This includes all the factors mentioned in Section 1.1.2 Present Day Issues, where corrosion resistance, difference in elastic modulus, and wear resistance become a part of the equation. Table 1 categorises the levels of biocompatibility, and what occurs at those levels with the implant.

Table 1: Biocompatibility levels with description

Biocompatibility level	Description
Bio-rejected	When the body reacts to the implant by encapsulating it in soft tissue, completely disallowing implant fixation. Implant can also cause a level of toxification and result in conditions occurring within 1-2 years of implant surgery [3].
Bio-tolerant	Similar to bio-rejected, body creates a soft tissue film around implant, but is much thinner and allows some level of implant fixation. This fixation is not permanent however, and may require revision surgery [3].
Bio-active	Bone strongly integrates with the implant's surface through formation of bone tissue around implant. This is the ideal result of implant fixation as the body has accepted the implant [3].

1.2 Aim

The aim of the thesis is to research, develop and test a titanium alloy that will meet the biocompatible level of 'bio-active', where implant acceptance and fixation will occur. This will be achieved through lowering the elastic modulus of the titanium alloy as close to human bone as possible, while ensuring that the alloy is also capable of producing a corrosion-resistant film that isn't toxic to the body. A level of wear resistance is also required to ensure metal particles do not enter the body.

2. Literature Review

The following section documents the findings on past literature that provide information relevant to the thesis. These findings include the methods to achieve better biocompatibility and what makes for an ideal implant alloy, potential candidates for said alloy and processing method.

2.1 Achieving the ideal alloy

The following sub-sections will cover the ideal alloy and its properties, as well as how to achieve it.

2.1.1 The required criteria

In any material-related industry, titanium is often considered one of the more ideal metals to be used in modern society. Commercially pure (CP) titanium has one of the highest strength-to-density ratios, and provides many other ideal mechanical properties such as corrosion- and wear-resistance, good balance between stiffness and ductility, and high endurance [3].
Another benefit of titanium is how changeable its original properties are based on the metal that is being alloyed with it. This stems from titanium's interchangeable microstructure, and allows it to essentially be customisable based on the required task.
For the purpose of the thesis and for the purpose of achieving biocompatibility, Table 2 will list the ideal titanium alloy with the desired mechanical properties.

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Table 2: Ideal properies for biomedical titanium alloy

Property	**Description**
Low elastic modulus difference with human bone	When two materials are in contact with each other, the material with the higher elastic modulus, or the stiffer material, will begin to cause surface damage and eventual fracture on the less stiff material [3]. In terms of implant and bone, the implant fractures the surface of the bone and causes bone cells to deteriorate and die [4]. Reducing the elastic modulus will allow the two materials to better interact without any form of plastic deformation.
High-corrosion resistance	All metals can be subject to corrosion depending on the environment they are placed in. Bodily fluids such as blood are considered one of the most aggressive environments a metal alloy can be subjected to, as blood contains corrosive molecules such as chloride ions and proteins [3]. Such circumstances demand a level of protection by the metal alloy, which comes in the form of a protective oxide film that prevents direct attack to the surface of the alloy [4]. Note that not all corrosion-resistant films are friendly to the human body, as is the case with Ti64.
High-wear resistance	Wear is the constant deterioration of a material from extended use, resulting in the release of residue or particles from the surface of the material [3]. In the case of titanium implants, the released particles into the bloodstream can cause levels of toxicity and allergic reactions. Increase in surface hardness can prevent wear [3].
Non-toxic	The metal must be non-toxic within human environment such as vanadium in the case of Ti64 [3].
Small level of porosity	Titanium alloys with small levels of porosity allow bodily fluid to pass through interconnected pores, which grants the implant better fixation and acceptance within the body [5]. Large isolated pores should be avoided however, as they can drastically reduce the strength of the alloy and localized corrosion due to excessive pore absorption [5].

2.1.2 Common methods

Titanium has two main allotropic forms:

- Its natural state at low temperature is a close packed hexagonal crystal lattice structure (HCP), which is often referred to as an α-dominant state [3].
- At higher temperatures above 880°C, it develops a body centered cubic lattice structure (BCC), which is termed as a β-dominant state [3].

The difference in the two structures is their level of compactness, which defines the stiffness of the titanium, or the elastic modulus [3]. Due to this, beta-stabilisation of titanium alloys is the common method for reducing the elastic modulus.

In order to achieve beta-dominance, it is insufficient to merely heat a titanium alloy to above 880°C and allow it to cool for application. When examining the microstructure of CP titanium past 880°C in Figure 1, it can be seen that there is a large presence of beta. However, when the same sample is allowed to cool down and examined again, it can be seen a small percentage of that beta from before is retained and the majority of the microstructure has returned to alpha.

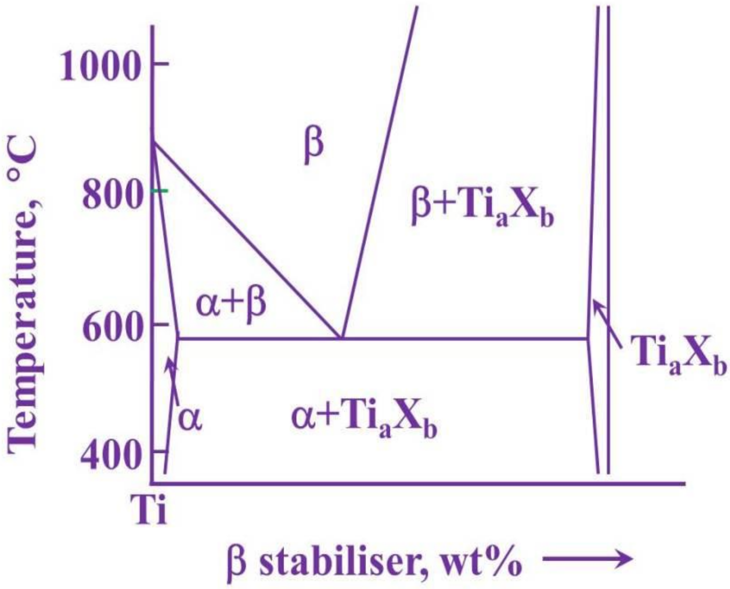

Figure 1:Phase diagram of a Ti-X, where X is a known beta-stabilizing metal [6]

As can be seen in Figure 1, if CP titanium is used with no weight percentage of a beta-stabilising metal, it will remain in the alpha region no matter its temperature. Yet this outcome can be changed with the addition of a beta-stabilising metal, which will allow the titanium alloy to remain in the beta phase, or α+β phase, even when cooled down. It should also be noted from Figure 1 that the beta transus temperature (the temperature which titanium becomes beta phase) is reduced with the addition of a beta-stabilising metal.

Thus, to achieve a beta-dominant titanium alloy, a beta-stabilising metal must be alloyed with titanium to not only increase the level of retained beta, but to also lower the beta transus so that beta-dominance can occur sooner.

Another well-known method to reducing elastic modulus is the introduction of porosity. This is mainly due to the porous structure lowering the overall density of the metal, resulting in a less brittle material [5].

Pores are often regarded as undesired defects resulting from the processing method, usually from powder sintering. They are undesired because they often lower mechanical properties such as yield strength, and even introduce levels of impurity into the alloy [4]. In extreme cases, if the pores within the alloys were to collapse, densification occurs which causes the stress of the alloy to rise exponentially [5].

Although it is often considered a defect, small levels of porosity that are controlled through type of alloying metal used and the addition of pore-making substances, can greatly benefit a titanium alloy. Not only does this come in the form of lower elastic modulus, but as mentioned in Section 2.1.1 The required criteria, it allows for the blood cells to circulate through the implant and grant better acceptance with adjacent bones [5].

2.2 Candidate metals

This section covers a list of metals to alloy with titanium that are believed to potentially meet the criteria set in Section 2.1.1 The required criteria, based on past literature.

2.2.1 Niobium (Nb)

Niobium is a rare metal often found in mineral columbite. It is primarily used in aerospace and civil application due to its ability to increase an alloy's strength at low temperatures [7].

Benefits:

- Lubricating properties of Nb_2O_5 provide high wear-resistance [3].
- Rich oxide film produced from niobium provide high corrosion-resistance on the alloy's surface, which is stable and non-toxic within the body [3].
- Known to increase beta phase by weakening alpha peaks, and thus lower the transus temperature of the alloy [3].
- Introduces small level of porosity, which is controlled by processing temperature [3].
- At approximately 30 wt% niobium, beta phase has been known to be entirely retained in the microstructure even after complete cooling [3].

Issues:

- Niobium has been known to lower compressive and yield strength when its content is increased in the alloy [3].

- Due to the limited number of natural deposits and the difficulty of minerally extracting it, niobium is classified a critical material, making it one of the most expensive metals for commercial purchase [8].
- Increases the melting point of titanium, therefore increasing the required heat and energy for processing [3].

2.2.2 Tantalum (Ta)

Tantalum is a rare metal found in mineral columbite-tantalite, which is the same mineral rock that niobium is found in. Its high strength makes it optimal for aerospace application, while its high capacitance makes it ideal for electrical appliances [9].

Benefits:

- Known to be biocompatible due to no toxicity [5].
- The Ta_2O_5 passive produced by tantalum strengthens the TiO_2 passive film on the surface of the alloy, increasing both corrosion- and wear-resistance [3].

Issues:

- Although it is classified as a beta-stabiliser, tantalum appears to be the only beta-stabiliser that increases elastic modulus rather than reduce it. This seems to stem from how a Ti-Ta alloy with high content of Ta begins to behave more like pure tantalum, which has a higher elastic modulus than CP titanium [3].
- Similar to niobium, tantalum is classified a critical metal due to its scarcity and difficulty to minerally extract [8].

2.2.3 Manganese (Mn)

Ranked as the fifth most abundant metal, manganese is found in most deposits around the world. Although extremely brittle in its pure form, manganese provides extra strength and wear-resistance to alloys that require it [10].

Benefits:

- Abundance of metal makes it highly available and low in cost [10].
- Prevents osteogenesis, a genetic disorder that causes bones to become brittle [11].
- Titanium alloys with approximately 13 mass% were found to demonstrate low levels of cytotoxicity [11].
- Strong compressive properties [11].
- When compared to Ti64, Ti-Mn demonstrates higher hardness and lower elastic modulus [11].

Issues:

- Beta-stabilisers have been known to increase elastic modulus when the alloying metal's content is too high (approximately around the 40% and above area). However, based on previous literature, this increase in elastic modulus phase occurs at much lower compositions of 9-10% [11]. The lowest possible elastic modulus occurs at Ti-8Mn, with a value of 87GPa.
- Due to the irregular shapes of manganese powders, sintering tends to cause large isolated pores within the alloy, which greatly reduces yield strength and increases chances of localised corrosion [11].
- Ductility decreases steadily as manganese content increases, making the implant less able to handle bending-type stresses [11].

2.2.4 Molybdenum (Mo)

Molybdenum comes from an ore called molybdenite, which is processed into its oxide form and then later reduced into its metal form. It has a high melting point and is used in alloys to increase yield strength, hardness, electrical conductivity and corrosion-resistance [12]

Benefits:

- Molybdenum's rich oxide film increases the corrosion-resistance of the TiO_2 film on the surface of the titanium alloy, which is also stable within the human body [3].
- Increases ductility in compression, with deformation rates reaching up to 50% without any indication of cracking or other forms of plastic deformation [5]
- Anodic oxides formed on the implant's surface benefit from improved stability and result in lowering potential for chemical reaction [4]
- Delays densification of titanium alloy due to low diffusion rate of molybdenum atoms and stronger porous structure [4].

Issues:

- Increase in molybdenum content has led to slight decline in compressive and yield strength [13].
- The quantity and structure of pore development caused by molybdenum causes melting point of titanium alloy to increase substantially, resulting in increased difficulty when processing or machining [3]

2.2.5 Iron (Fe)

Iron is the most widely used metal throughout the world, finding its purpose with 90% of industries such as manufacturing, civil engineering, automotive, and aerospace applications. It is primarily used as a base metal for the requirement of strength and hardness [14].

Benefits:

- High diffusion rate with titanium, resulting in better alloying in processes such as sintering [15].
- Strong beta-stabiliser as it greatly slows the phase change of beta back to alpha in its cooling process, thus retaining larger amounts of beta [15].
- Mechanical properties of Ti-Fe alloys can be further adjusted by cooling schedule and/or heat treatment more so than other titanium alloys [15].
- Iron content and beta content share an increasing linear relationship [15].
- Increasing iron content also increases hardness [15].

Issues:

- Increasing iron content beyond 3 wt% causes yield strength and elongation to reduce by a substantial amount [15].
- Laths in the microstructure demonstrate that alpha phase of Ti-Fe is more corrosion-resistant than beta or $\alpha+\beta$ phase [15].
- Requires high temperatures of 740^{o}C and above to develop and maintain desired levels of beta phase [15].

2.3 Manufacturing methods

The biomedical industry utilises a range of processing methods based on the desired mechanical properties for the alloy, as well as a manner that is cost-effective. It is common knowledge among the material industry that although the metals in an alloy dictate the main mechanical properties it will inherit, it is the manufacturing method that can either enhance the desired properties, or minimize the undesired traits.

Due to the nature of the biomedical industry, where very specific shapes and sizes are required, common manufacturing methods such as die casting are inviable as they are mostly fixed to one shape and size [3]. The difficulty and costs of obtaining the metal and liquifying it are also high. It is from this that the concept of powder metallurgy (PM) was born, where metals could be powdered and alloyed together at a temperature below their respective melting points [3]. The following sub-sections cover the different manufacturing methods that can be used with PM.

2.3.1 Cold sintering

The most common form of sintering and most used with PM, cold sintering alloys metals through fused metallic bonding when the temperature is just below the melting point of the metal with the lower melting point. The lower temperature requirement benefits from lower cost, while the fusing method grants a larger variety of potential geometries and sizes [3].

2.3.2 Gas atomized

The powdered metals are first heated with an inert gas in a water-cooled copper crucible. The heat metal powders are then poured into an induction heated nozzle and atomized with high-pressure argon gas. This process results in a purer and more precise alloy, increasing chances of getting desired mechanical properties. Unfortunately, it is more expensive and production rate is lower compared to other methods [16].

2.3.3 Metal Injection Molding

Metal injection molding involves combining the metal powders with a binder (often wax or a thermoplastic), where this mixture is termed the feedstock. The feedstock is blended through shear force, and is then cooled into a granulate material. The binder component is then removed by either thermal extraction or solvent processing, and the remaining material is then sintered to required specifications in a controlled-atmosphere furnace. This process favors economies of scale, with a high degree of freedom with geometry selection and high reproducibility. Drawback is that it is limited to small sizes [17].

2.3.4 Electromagnetic sintering

During the sintering stage of the metal, electrical current is driven through the metals powders to develop an electromagnetic charge. This charge enables the powders to alloy faster and more securely, which lowers the probability of impurities and undesired pores [18].

2.3.5 Common Issues of PM

Similar to any manufacturing method, methods that utilise PM are subject to defects and faults that can, and in most cases, will affect the alloy product. These include:

- Agglomeration: During the mixing process of the metal powders, it is common for traces of the powder to stick to the side of the mixing container. This can result in distortion of alloy composition, leading to inaccurate analytical conclusions [19].
- Foreign particle staining: If care is not taken during the PM mixing stage and/or during the post-processing stage, foreign particles may cause alterations to the alloy. The most common of these incidents is the staining of an alloys surface with cleaning ethanol, which hinders the ability to properly analyse the alloy's microstructure.
- Undesired high level of porosity: If compaction is done incorrectly, or foreign particles contaminate the PM mixture, the probability of high levels of porosity is increased. This will result in the alteration of the alloy's mechanical properties such as greatly reducing the yield strength of it [4].

2.4 Evaluation process of potential alloying metal

In order to better analyze and define the effects different metals have on titanium, only one of the candidates from Section 2.2 will be chosen. To assist in the decision process, Table 3 will define the weighting of the criteria with explanation, while Table 4 will act as the final decision matrix. The weighting in Table 3 is defined as 1 being the least important and 5 being the most important.

Table 3: Criteria weighting with reasoning for candidate metals

Criteria	**Weight**	**Reason for weighting**
Low elastic modulus difference with human bone (LE)	5	Achieving lower elastic modulus is the primary goal of the thesis, being the most difficult criteria to fulfil. Ensuring as low elastic modulus as possible is also the best method to achieving biocompatibility, as implant integration depends heavily on the implant being similar to that of bone.
High corrosion-resistance (CR)	4	It is paramount that no corrosion takes place within the human body, as such contamination will like result in debilitating illnesses for the implant recipient.
High wear-resistance (WR)	3	Although most titanium alloys already have good wear-resistance, it would be a vigilant choice to increase it further to ensure the endurance of the implant for longer periods of time without any particle release into the body.
Non-toxic (NT)	4	Similar to the high corrosion-resistance criteria, any level of toxicity could result in illness or other adverse effects.
Small level of porosity (SP)	2	Small levels of porosity are not a required criterion, but rather a feature that better guarantees the integration of the implant with adjacent bones.
Cost (C)	4	Although not a primary objective of the thesis, the costs of metals should be considered as it will result in the long-term feasibility of future developed implants.
Reduction of other properties (RP)	4	As can be seen in Section 2.2 Candidate metals, alloying metals have also been known to reduce some of the mechanical properties of titanium, such as lowering yield strength and ductility. It is important to maintain such properties to a certain degree.

Most decision matrices also include a description of how a candidate can achieve a certain score based on the criteria. However, this is difficult to quantify with some criteria such as non-toxic and porosity level, resulting in the candidate scores of the decision matrix to be estimates based on the literature covered in Section 2.2 Candidate metals. Table 4 will outline the scores and weighted scores to conclude the deciding alloying metal. The lowest to highest goes from 1 to 5, and the metal with the highest score will be chosen.

Table 4: Decision matrix for candidate metals

Metals	**LE (5)**	**CR (4)**	**WR (3)**	**NT (4)**	**SP (2)**	**C (4)**	**RP (4)**	**Total score**	**Weighted total score**
Nb	4	3	4	3	2	1	2	19	72
Ta	2	5	2	5	1	1	2	18	70
Mn	3	4	2	4	1	4	4	22	87
Mo	3	5	4	4	3	3	3	25	93
Fe	4	2	4	2	1	5	2	20	78

2.5 Specific evaluation of research objectives

Utilising the literature research and decision matrix developed in Section 2. Literature Review, the chosen metal to alloy with titanium will be molybdenum. In terms of manufacturing method to produce the alloy, cold sintering will be used as it is the most cost and time effective compared to the other manufacturing methods.

Therefore, the re-evaluated aim of the thesis is to determine whether molybdenum can achieve the desired results of lowering the elastic modulus of titanium, while still maintaining all the other desired mechanical properties.

2.6 Thesis scope

Due to the potential complexity and scale of the thesis, it must be considered what should and should not be in the scope. This will allow for better focus on the factors that would reduce the elastic modulus of titanium alloy. Table 5 outlines the scope of the thesis.

Table 5: Thesis scope outline

In-scope	**Out-of-scope**
• Specific alloy composition • Compression testing • Microstructural analysis • Hardness testing	• Post-processing treatment • Complex alloy compositions (more than 2 metals) • Impact testing • Tensile testing • Fracture toughness testing • Surface conditioning • X-Ray inspection • Magnetic particle inspection • Corrosion testing

3. Methodology

The methodology section outlines the preparations for alloying, the manufacturing process and the post-processing procedures in developing research samples of titanium-molybdenum (Ti-Mo). The section will then be finalised with individual sub-sections that describe the mechanical testing conducted on said samples, undergoing compression testing, Vickers hardness testing and microstructural analysis.

3.1 Alloy composition

It was noticed in many past literatures that when titanium alloys with the same alloying metal, but different compositions were tested, the incremental increase in composition was very low. Many would start at Ti-2X wt%, and only increase by 2% each time. Obvious differences could be noticed with each increase, but to a very small degree. In order to notice more immediate changes within a titanium alloy, the incremental increase in composition will be greater. The three compositions will be Ti-5Mo, Ti-10Mo and Ti-15Mo. Samples of pure titanium will also be prepared to be used as a point of reference and comparison during the result analysis stage.

Each sample composition would weigh between 5-7 g, with the first composition being 95% titanium and 5% molybdenum, all the way up to 85% titanium and 15% molybdenum. Once each sample composition was prepared, two more of each were prepared to a total of 12 samples, including the pure titanium samples. All sample powders were then placed in plastic containers to be thoroughly mixed in the powder blending device. An image of the PM blender is provided in Figure 2.

Figure 2: Image of powder blending device

After all samples (except pure titanium) were thoroughly mixed for an hour, each mixture was then carefully poured into a compacting mould, which was then placed into a PM compacter and pressurised at 550 MPa (180 Psi). An image of the compacter used can be viewed in Figure 3.

Figure 3: Image of PM compacter used for thesis

Once all nine samples were compacted to acceptable firmness, they were then placed into a sintering furnace at 1300^{o}C for 3 hours, with a heating rate of 4^{o}C/minute. Samples were then left to cool at the same rate in the furnace until standard room temperature. An image of the sinter furnace used can be viewed in Figure 4.

Figure 4: Image of sinter furnace used for thesis

The reason samples are heated to 1300°C is because the melting point of titanium is approximately at 1600°C, and during sintering it is ideal to reach a temperature hot enough to initiate diffusion between particles, but not too high that melting begins to occur. Molybdenum is the metal with the higher melting of 2623°C, making 1300°C sufficient to cause diffusion as well. An image of the samples after cooling is provided in Figure 5.

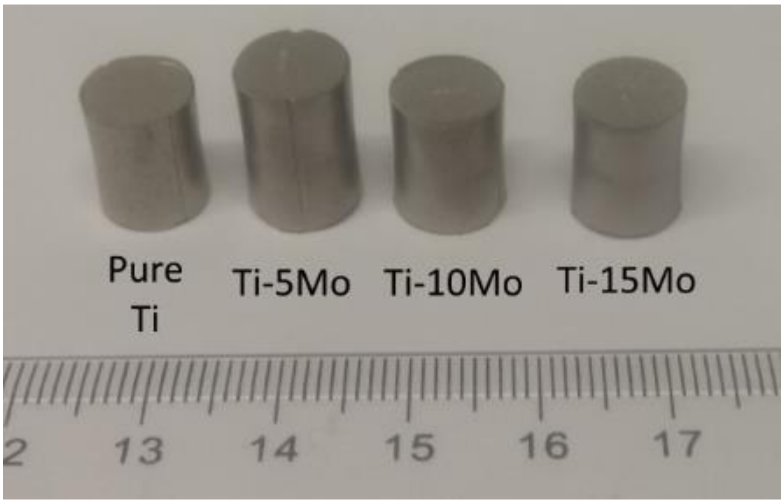

Figure 5: Image of samples after cooling

3.2 Microstructural analysis

One batch of samples were separated from the rest for the purpose of microstructural analysis. These samples were cut in half via diamond cutter and then embedded into a thermoplastic hot resin mount. An image of the machine used for hot resin mount is provided in Figure 6.

Figure 6: Struers hot resin mounting machine [20]

Once the hot resin mounting cooled down, the samples were then extracted for inspection. An image of the samples after hot resin mounting are provided in Figure 7.

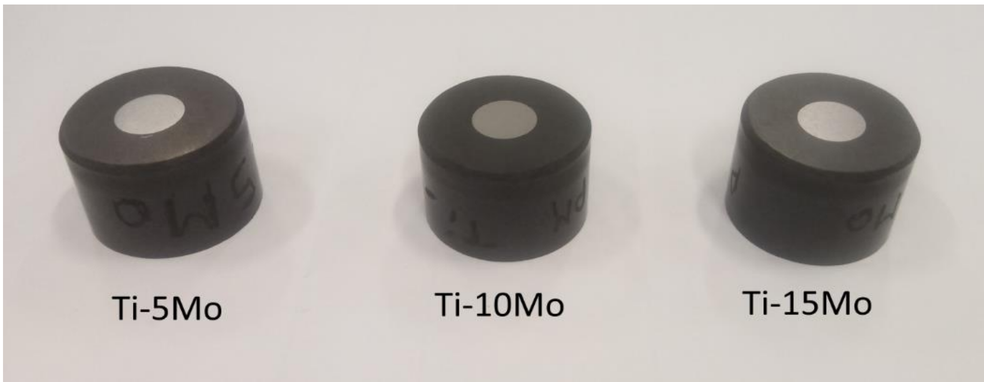

Figure 7: Samples after hot resin mounting

To ensure the clearest imagery possible during microstructural analysis, the samples then underwent a series of post-processing procedures. The first process involved grinding the samples with a Struers Rotopol grinder. An image of the grinder is provided in Figure 8.

Figure 8: Image of Struers Rotopol grinder

Table 6 outlines the grinding process parameters, where the samples were first grinded with 600 paper.

Table 6: Procedural outline of grinding process

Paper Quality	**Number of times sample undergoes process**	**Force (N)**	**Duration (minutes)**	**Sample holder rotation speed (RPM)**	**Disc rotation speed (RPM)**
600	1	20	1	150, anti-clockwise	300
1200	2	20	1.5	150, anti-clockwise	300
4000	2	20	1	150, anti-clockwise	300

After thorough inspection of the surfaces of the samples, with no visible scratches, the samples were then rinsed with ethanol and saline water, before being submerged in ethanol and placed in ultrasound cleaning bath for 5 minutes. Once second inspection was complete and no visible contaminants found, the samples underwent polishing procedures. The Struers Rotopol grinder was still used for polishing with the same parameters, except grinding plate was changed for a polishing magnetic plate. Table 7 below outlines the parameters for polishing.

Table 7: Procedural outline for polishing process

Disc surface	**Duration (minutes)**	**Polishing liquid**	**Interval between polishing liquid addition (seconds)**
MD-Chem	Up to 10	OP-S + 10 vol. % H_2O_2	30

Upon completion of polishing, samples were again rinsed in ethanol and left to dry for one hour. A final inspection took place to ensure that all samples were defect-free, before initiating etching procedures. The delicate procedure is conducted at a ventilated cabinet, where hydrofluoric acid was applied to the surface of all the samples for four seconds. Immediately after the four seconds, the samples are submerged in ethanol for 5 seconds, before being placed in a petri dish to dry for another hour. The samples were then ready for complete microstructural analysis.

Each sample examined had images taken in three different areas through microscopic image photography. This was to allow for better understanding of the microstructure through a wider scope of imagery to examine.

3.3 Compression testing

The remaining samples underwent compression testing using a compression tester. Standard safety procedures were carried out during testing, ensuring safety shield was in place and that all research members were at least 1 metre away from compression machine during testing. An image of the compression machine used is provided in Figure 9.

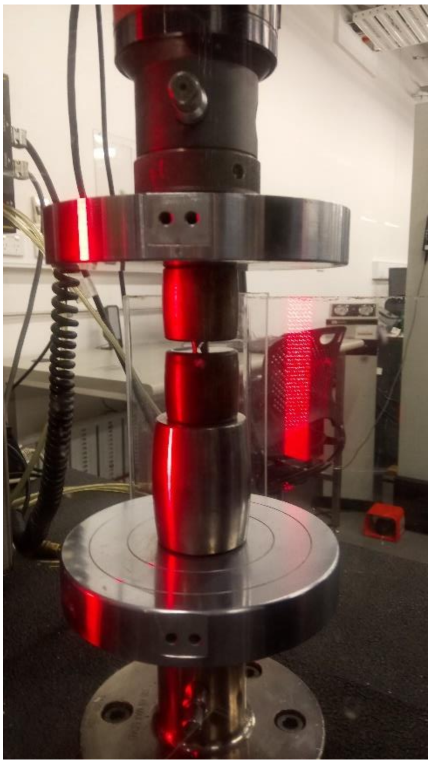

Figure 9: Image of Compression machine

An image of a sample after and before compression testing is provided in Figure 10.

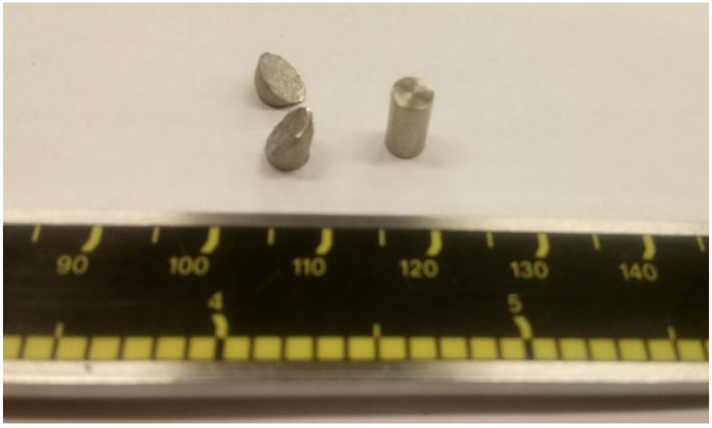

Figure 10: Ti-5Mo samples after and before compression testing

All data was recorded and collected from compression analyser program via computer console connected to the compression machine.

3.4 Vickers Hardness testing

It was not initially planned to conduct Vickers hardness testing due to complications with the Struers Vickers hardness tester at the time, however the machine was eventually calibrated by university staff and was fit for duty.

New samples were developed for each composition including pure titanium, and then prepared the same way the samples were for microstructural analysis. The samples would then be tested in a newly calibrated Struers Vickers hardness tester. An image of the Struers Vickers hardness tester is provided in Figure 11.

Figure 11: Struers Vickers hardness tester

Each sample underwent 15 trials of indentation and Vickers scanning, with the values of Vickers hardness for each trial being recorded manually in excel. Care was taken with every trial by ensuring the indentation was as close to diamond shape as possible. If diamond shape was deemed too unusual or irregular, the trial was redone. An example of how an indentation should look is provided in Figure 12.

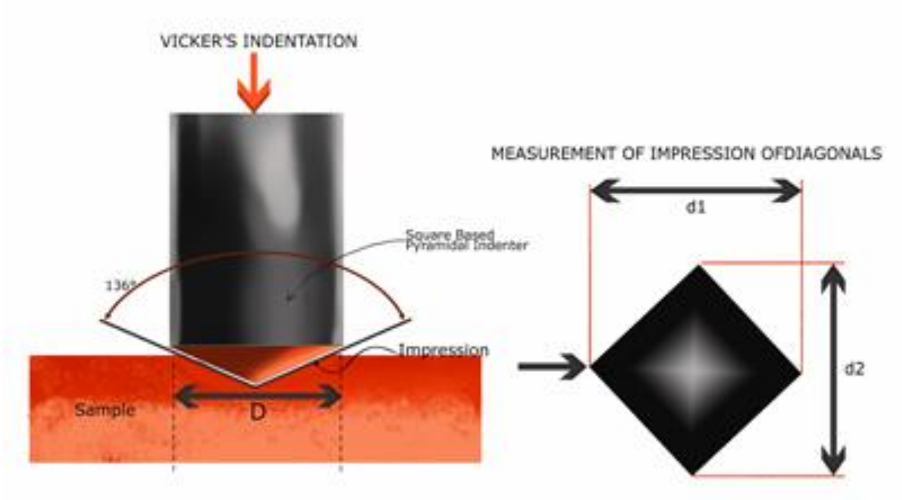

Figure 12: Vickers hardness indentation process [21]

The specifications of the Vickers hardness testing are provided in Table 8.

Table 8: Vickers hardness specifications

Load (Newtons)	**HV**	**Time (seconds)**	**Conv. HRC**
2.942	0.3	12	28.2

4. Results

The results sections demonstrate the findings over all testing and analysis procedures outlined in Section 3. The section will only display the findings from the microstructural analysis, compression testing and Vickers hardness testing, with only minor commenting to highlight any abnormalities.

4.1 Microstructural analysis results

For each composition, only one out of three images will be displayed in Section 4.1 to demonstrate the most significant findings. Refer to Appendix A: Images of microstructural analysis for other images.

4.1.1 Imagery at 20x magnification

The imagery displayed in the following sub-section are all at 20x magnification. Figure 13, Figure 14, Figure 15 and Figure 16 show the microscopic imagery for pure titanium, Ti-5Mo, Ti-10Mo and Ti-15Mo respectively.

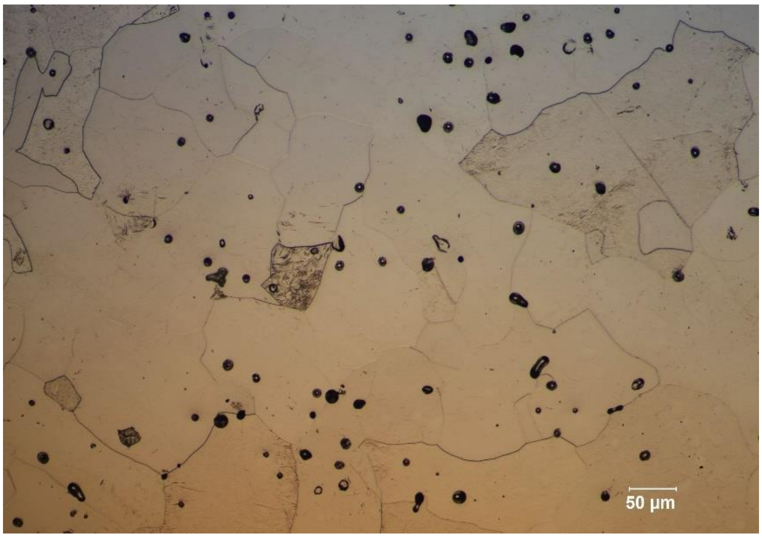

Figure 13: Pure Ti microscopic image at 20x magnification

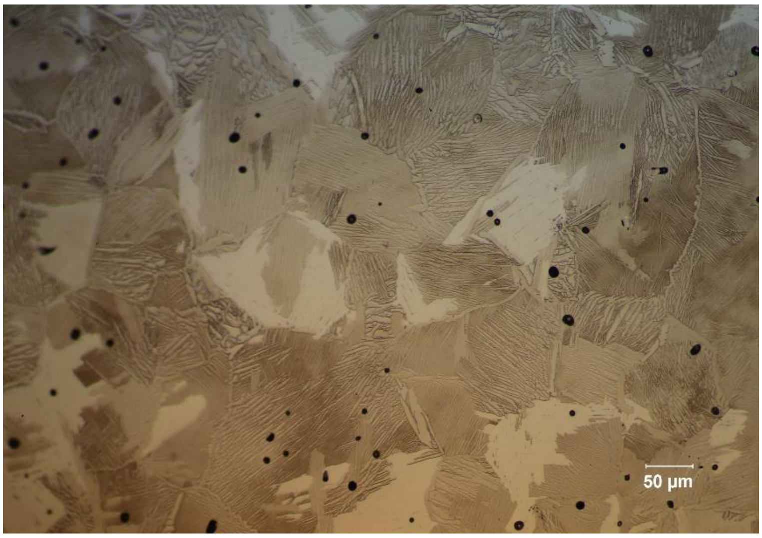

Figure 14: Ti-5Mo microscopic image at 20x magnification

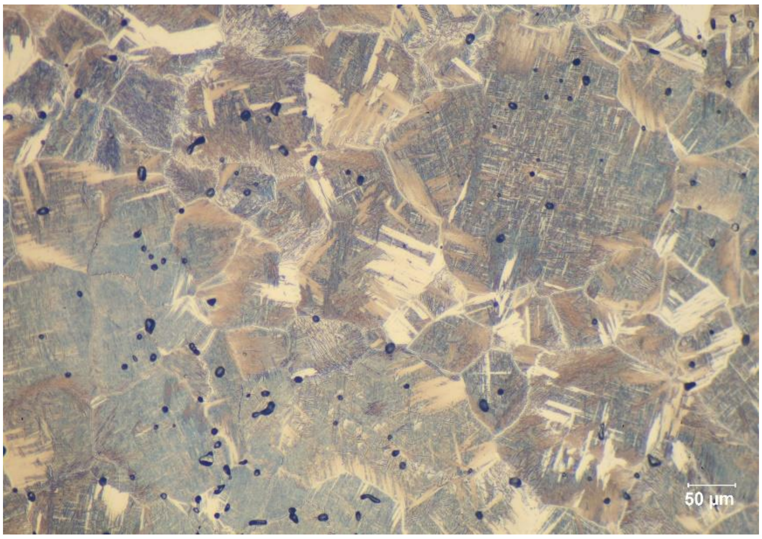

Figure 15: Ti-10Mo microscopic image at 20x magnification

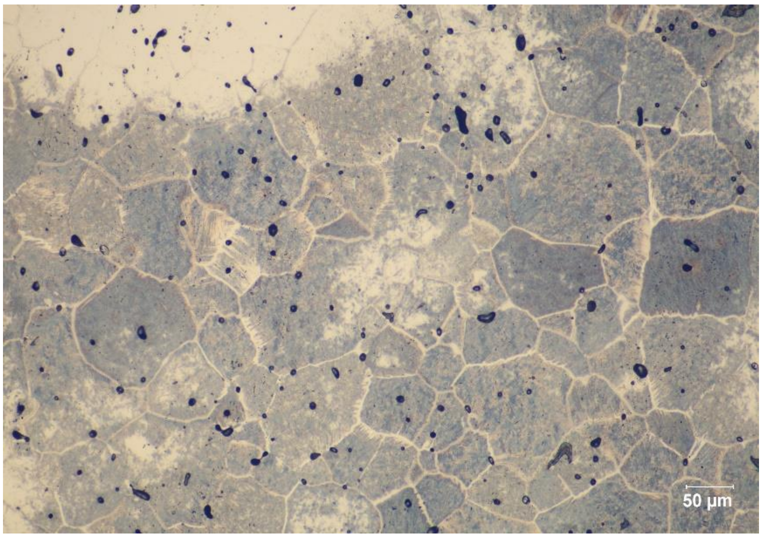

Figure 16: Ti-15Mo microscopic image at 20x magnification

4.1.2 Imagery at 50x magnification

The imagery displayed in the following sub-section are all at 50x magnification. Figure 17, Figure 18, Figure 19, Figure 20 show the microscopic imagery for pure titanium, Ti-5Mo, Ti-10Mo and Ti-15Mo respectively.

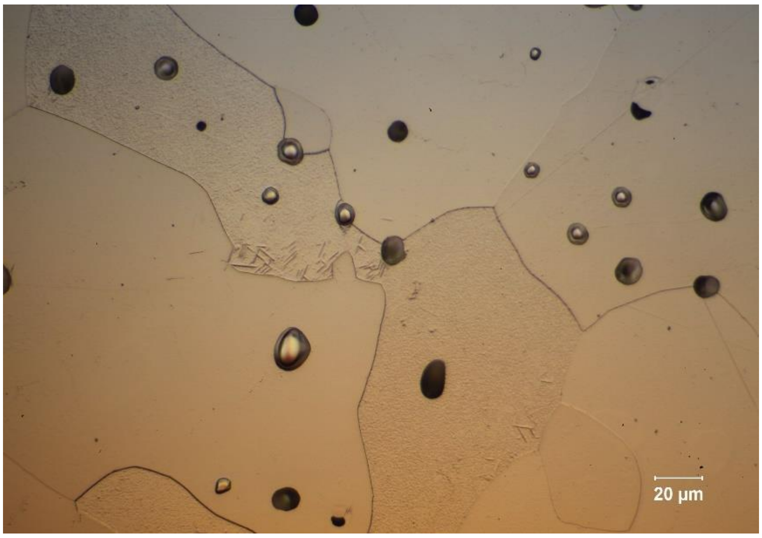

Figure 17: Pure Ti microscopic imagery at 50x magnification

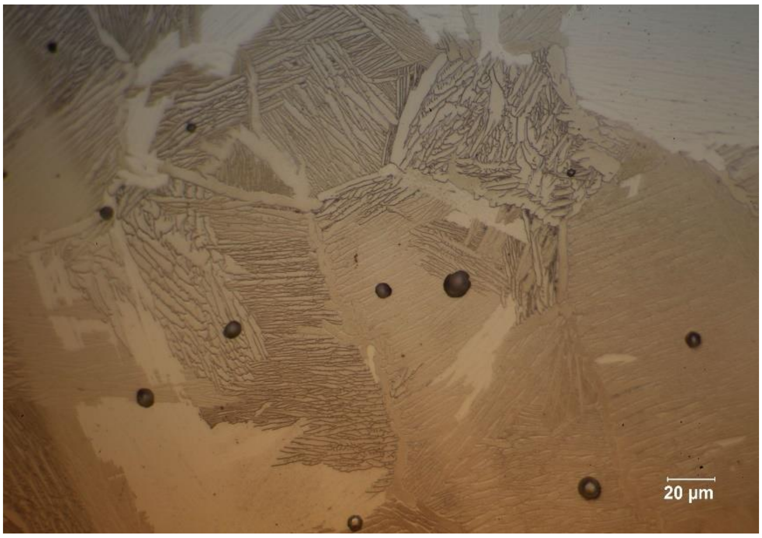

Figure 18: Ti-5Mo microscopic imagery at 50x magnification

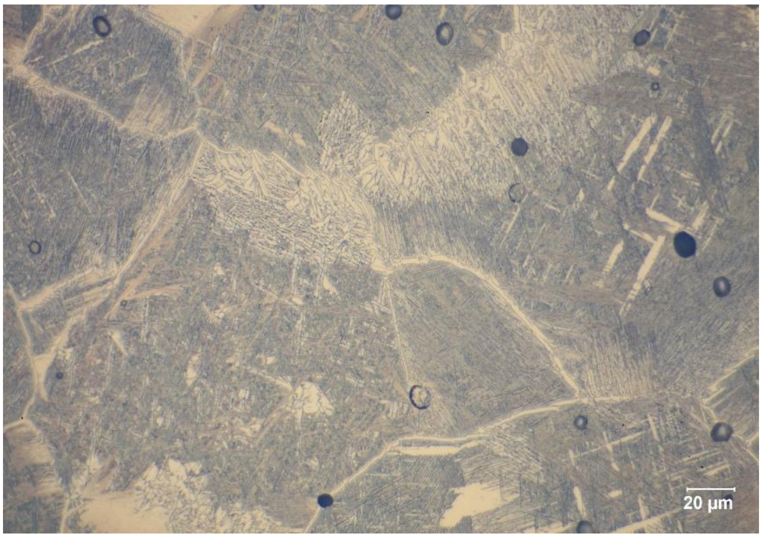

Figure 19: Ti-10Mo microscopic imagery at 50x magnification

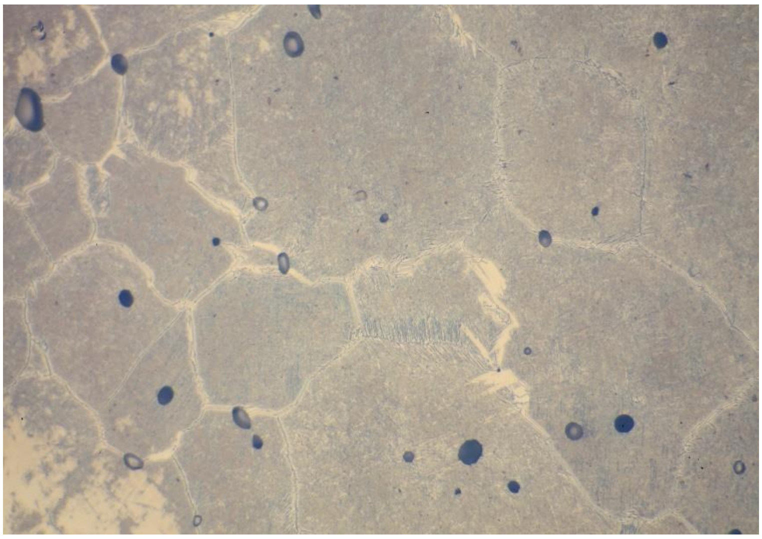

Figure 20: Ti-15Mo microscopic imagery at 50x magnification

4.2 Compression testing results

There were two main batches of samples for compression testing, and the results from both batches will be displayed next to each other based on their composition, with a number title to indicate which batch the sample belongs to. Figure 21, Figure 22, Figure 23 and Figure 24 demonstrate the stress-strain curves of pure titanium, Ti-5Mo. Ti-10Mo and Ti-15Mo respectively.

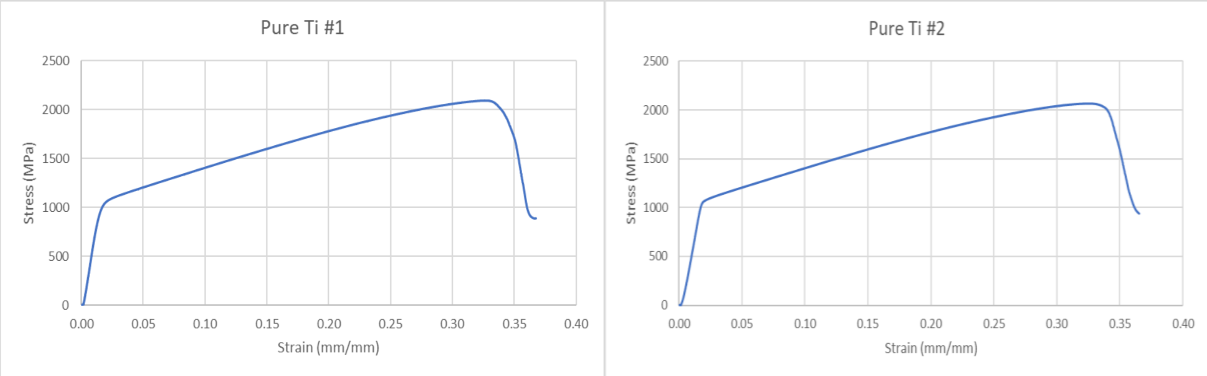

Figure 21: Pure Ti stress-strain curves

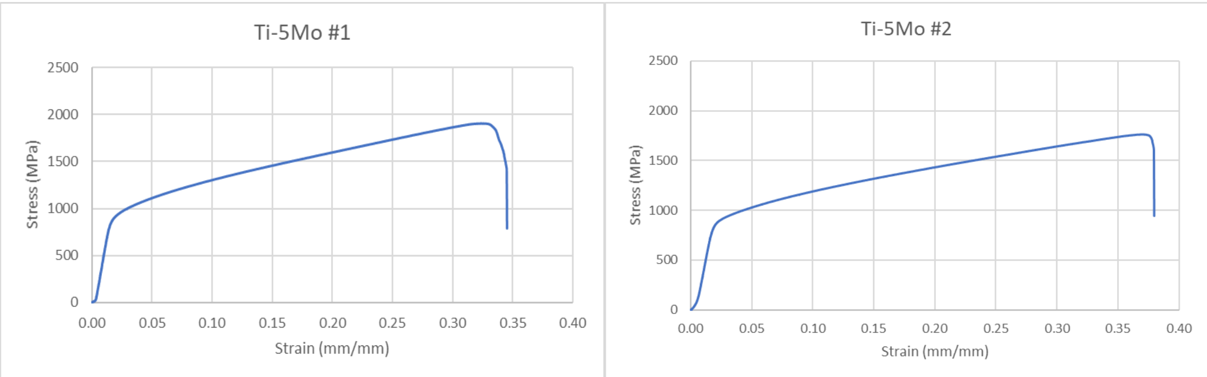

Figure 22: Ti-5Mo stress-strain curves

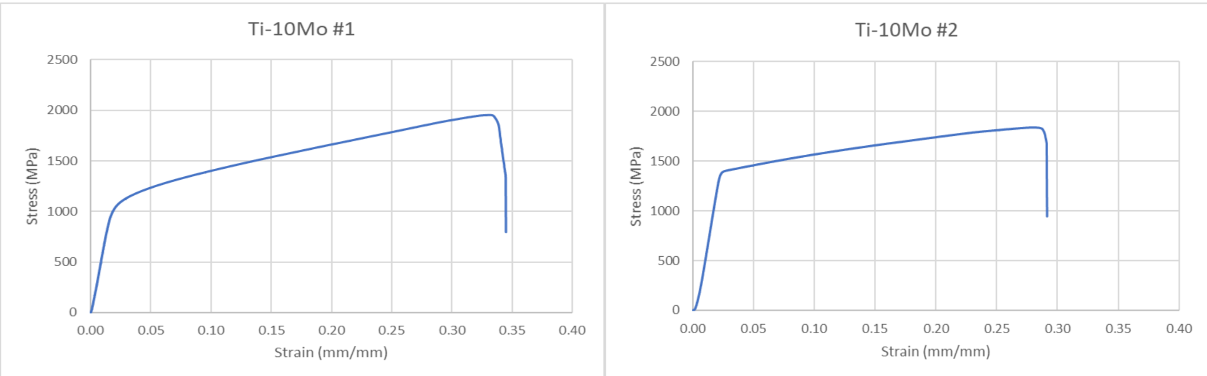

Figure 23: Ti-10Mo stress-strain curves

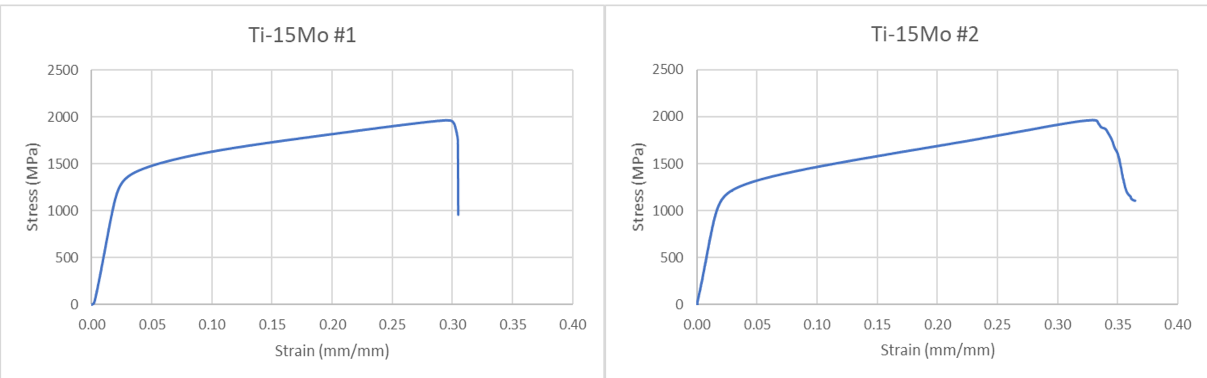

Figure 24: Ti-15Mo stress-strain curves

The elastic modulus of each sample was calculated by examining the average elastic modulus during the linear-elastic phase of deformation. The formula used for calculating the elastic modulus was Hooke's Law, displayed as Equation (1).

$$\sigma = \varepsilon E \tag{1}$$

Where:

- σ = stress (MPa)
- ε = strain (mm/mm)
- E = elastic modulus (GPa)

The average of the two sample batches based on alloy composition were then used and compiled in Table 9. Ultimate yield point was found based on the maximum stress on the curve and was then averaged as well.

Table 9: Average elastic modulus and yield point of each composition

Composition	Elastic Modulus (GPa)	Ultimate yield point (MPa)
Pure Ti	46.59	2079.05
Ti-5Mo	35.66	1834.56
Ti-10Mo	47.57	1896.07
Ti-15Mo	52.56	1964.89

4.3 Vickers hardness results

Table 10 displays the results from the Vickers hardness testing, which were the averages of all 15 trials from each composition. Refer to

Appendix B: Vickers hardness test results for complete data.

Table 10: Vickers hardness results

Composition	Vickers Hardness (HV)
Pure Ti	288.22
Ti-5Mo	301.58
Ti-10Mo	335.87
Ti-15Mo	391.60

5. Discussion

The discussion section will talk about the results provided in Section 4, explaining any discrepancies in the data or any potential anomalies. Discussion will also include comparison to past literature results, and drawing upon similarities to better understand findings.

5.1 Microstructural analysis

In order to establish what each microstructure represents, images taken from Section 4. Results will be compared to images taken in previous literature, with explanation as to what each part of the microstructure is, and how it affects the alloy as a whole. Each comparison image has the past literature image on the left, and the current thesis image on the right.

5.1.1 Pure Titanium microstructural analysis

When examining Figure 13 and Figure 17, the majority of image is occupied by bright regions with small sections being comprised of shaded regions. The bright regions are labelled as equiaxed alpha, and represents alpha-dominance in an alloy [22]. It is the common microstructure for unalloyed titanium, which as mentioned in Section 2.1.1 The required criteria, is naturally in an alpha-dominant state. The shaded regions are termed as acicular alpha, which is a phase that occurs as a minority within alpha-dominant alloys, but develops as beta-presence increases.

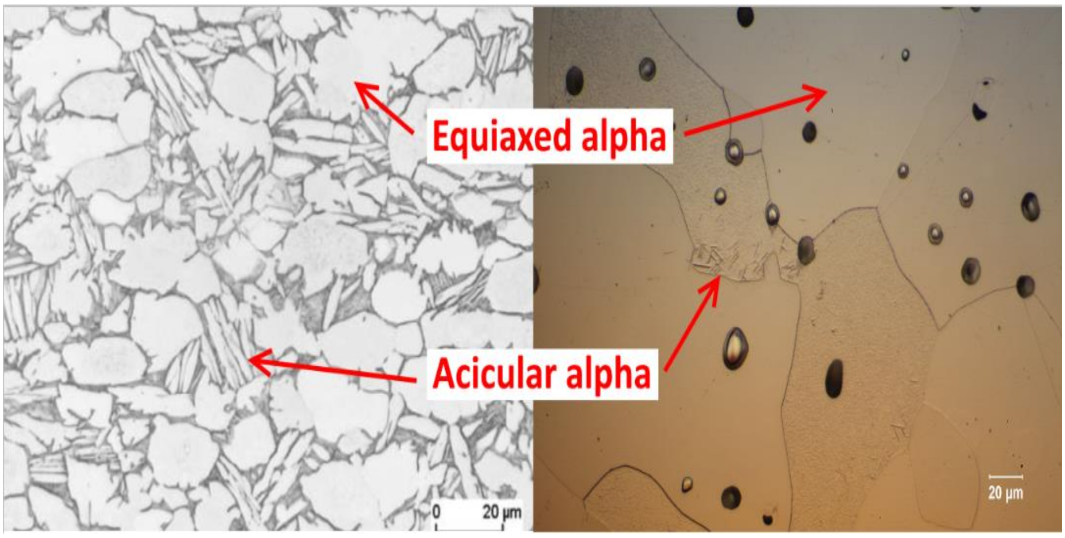

Figure 25: Pure Ti phase classification comparison with Donachie's literature [23]

5.1.2 Ti-5Mo microstructural analysis

With the inclusion of molybdenum in the alloy, it can be seen in Figure 14 and Figure 18 that the microstructure undergoes drastic change. This state is termed ‘alpha plus developing beta’. The bright regions of equiaxed alpha have almost disappeared, with the remaining alpha being thin plates. However, it can be noted that between those alpha plates, beta development has begun to occur.

The beta in Ti-5Mo initiates as dark lines between alpha plates, with varying levels of darkness depending on the density of alpha plates in a specific area. Examining Figure 26, areas with more densely clustered alpha plates such as Exhibit A are surrounded by lighter shades of beta, while areas such as Exhibit B with fewer alpha plates are bounded by darker beta shades.

Beta grain boundaries have also begun to develop with the addition of molybdenum, appearing as borders around sections of clustered alpha and beta. These boundaries are classified as unrefined, primarily due to the arbitrary shapes and thicknesses they display at different points. Looking closely at Figure 26, Exhibit C demonstrates a thick but short, finger-shaped boundary while Exhibit D demonstrates a slenderer pole-shaped boundary.

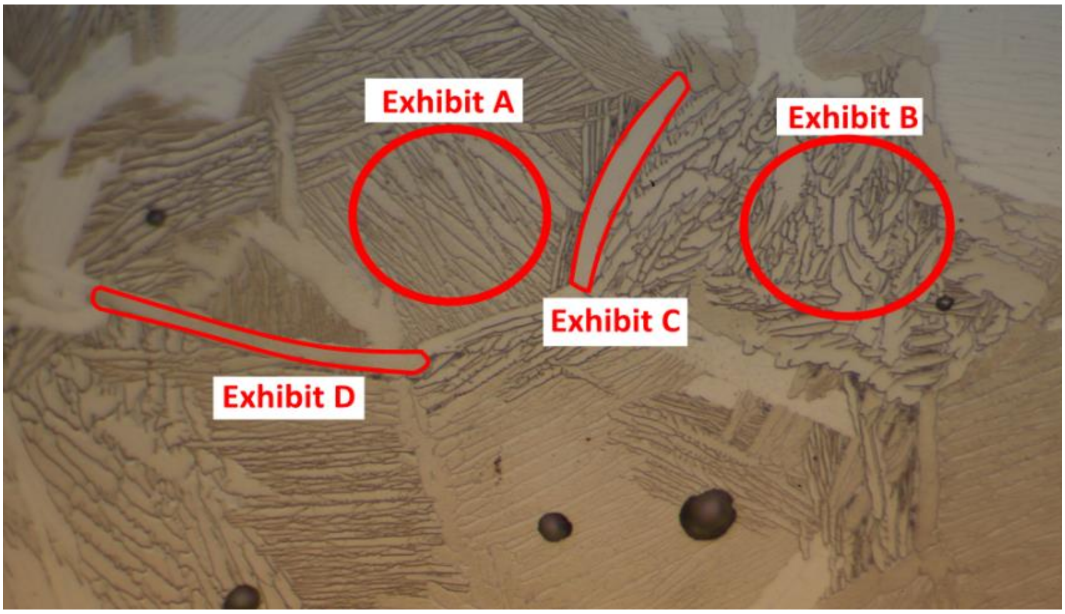

Figure 26: Exhibit display of beta initialisation in titanium alloy

For a comparison to Donachie's phase classification, refer to Figure 27.

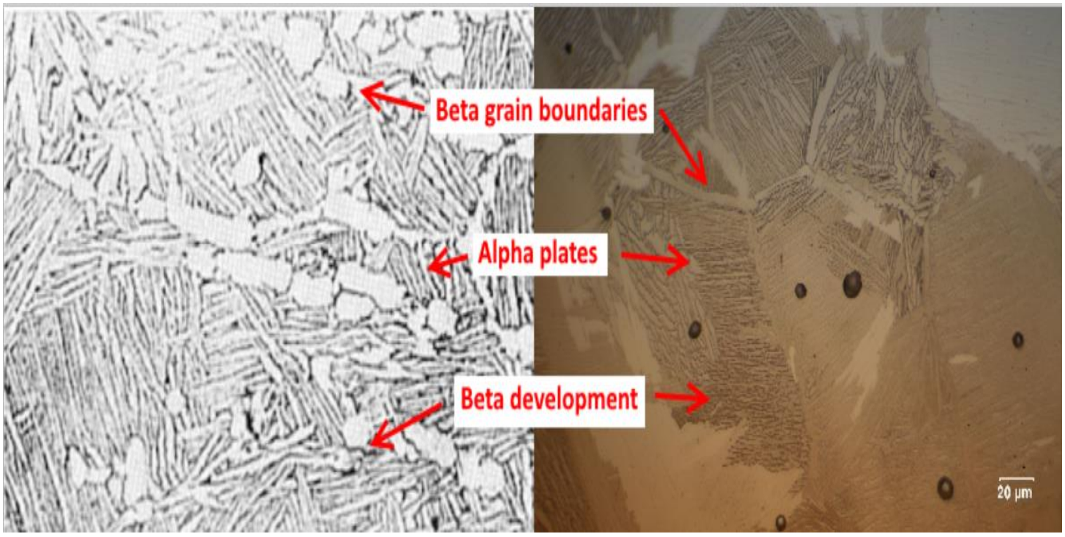

Figure 27: Alpha plus beta alloy phase classification comparison with Donachie's literature [23]

5.1.3 Ti-10Mo microstructural analysis

At 10% molybdenum content, the dark beta regions that punctuated the alpha plates have now overtaken the majority of the sections within the microstructure, turning developing beta into transformed beta. It should also be noted from Figure 19, that the brighter equiaxed alpha from pure titanium has almost been completely replaced by the greyer acicular alpha, demonstrating that the alloy has already transformed from its original HCP state to BCC state mentioned in Section 2.1.2 Common methods.

Another important factor in the development of beta phase is the refinement of beta grain boundaries bordering the different sections of the microstructure. Compared to Ti-5Mo, the grain boundaries are more characterised and refined through their consistent uniform shapes and thicknesses. This is often termed 'prior grain boundaries'. For a comparison to Donachie's phase classification, refer to Figure 28.

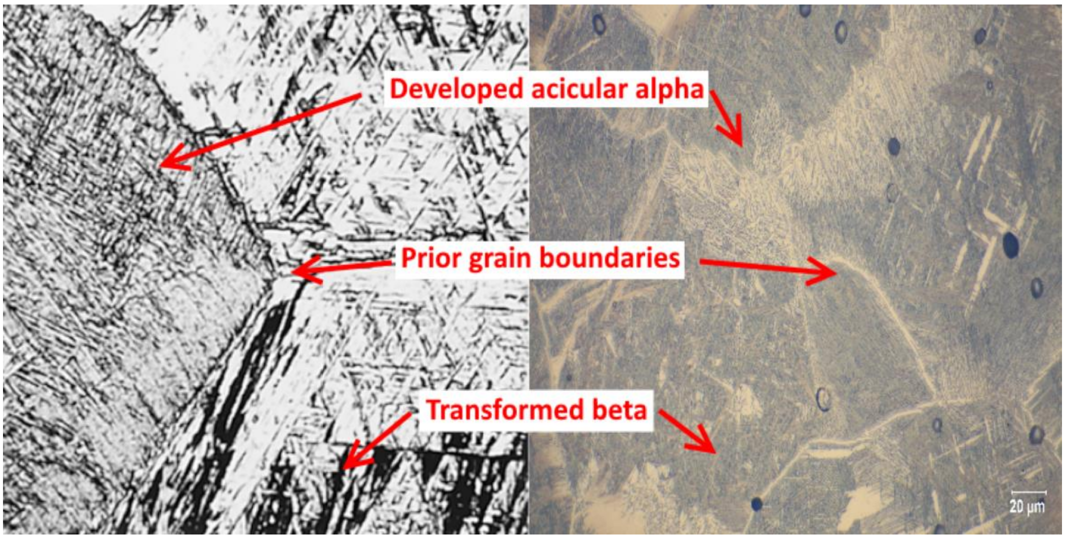

Figure 28: Transformed beta alloy phase classification comparison with Donachie's literature [23]

5.1.4 Ti-15Mo microstructural analysis

Finally, at Ti-15Mo, using Figure 20 as evidence, beta-dominance has been completely achieved within the titanium alloy. The dark sections of beta in Ti-10Mo have become dark beta regions that occupy almost the entirety of the microstructure, with very minor alpha phase remaining as bright packets. For a comparison to Delvat et al's phase classification, refer to Figure 29.

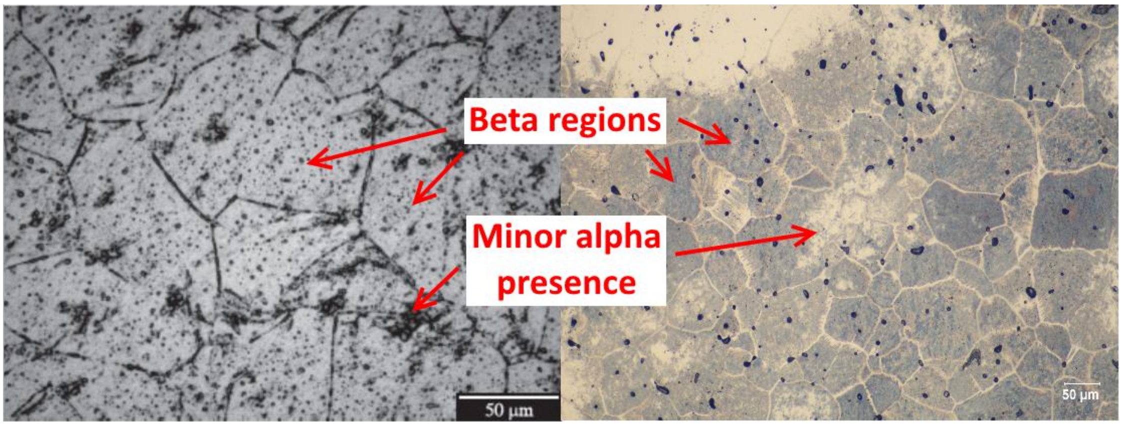

Figure 29: Beta-dominant alloy phase classification comparison with Delvat et al. [6]

5.1.5 Summary of microstructural analysis

As could be seen throughout Section 5.1 Microstructural analysis, comparisons with past literature were made to ensure proper classification of phases were made. Based on those comparisons, the increase in molybdenum content did in fact increase beta presence within the titanium alloy samples. This could be examined from the gradual increases in dark regions, while the brighter alpha regions decreased, as well as the addition of the beta grain refinement becoming more characterised and refined. The transformation from equiaxed alpha to acicular alpha also demonstrates a level of phase change, as acicular alpha normally occurs with increase in beta development.

5.2 Compression testing

Using the discussion made in Section 5.1 Microstructural analysis, results from compression testing can be directly related to the actual beta development with increasing molybdenum composition. For easier comparison between different alloy compositions, Figure 30 and Figure 31display the stress-strain curves of the first and second test batch of samples respectively. All samples ended in ductile failure.

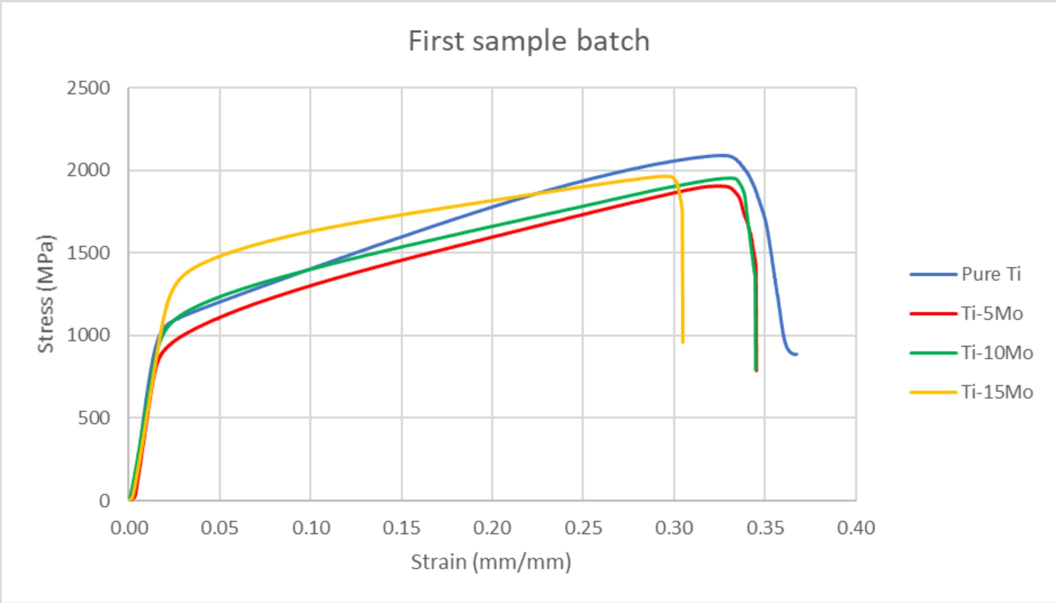

Figure 30: Stress-strain curve comparison of first test batch of Ti samples

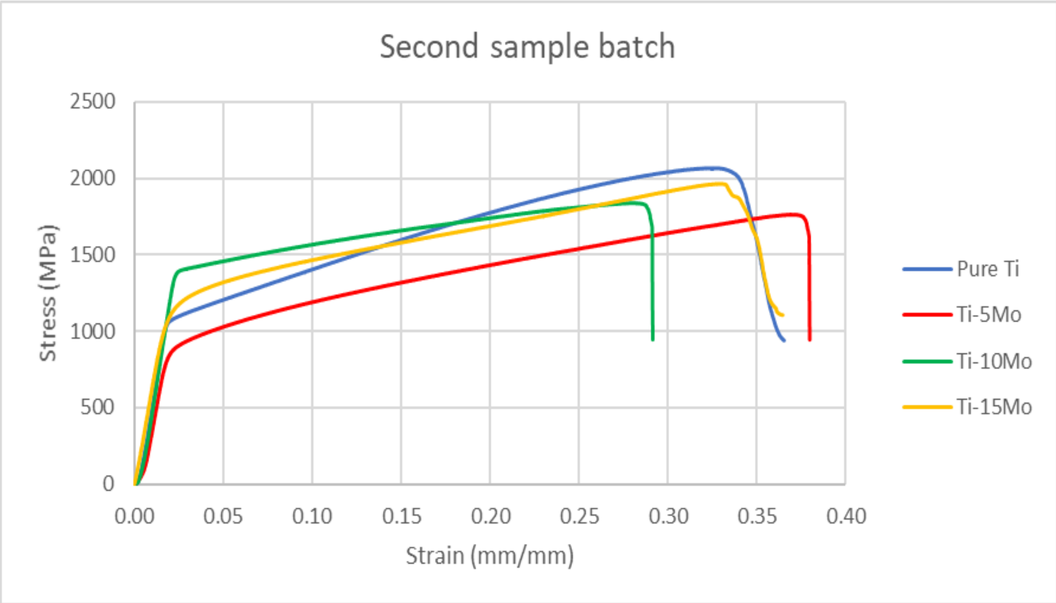

Figure 31: Stress-strain curve comparison of second test batch of Ti samples

5.2.1 Mechanical property interpretation

As the elastic modulus is directly proportional to stress, it is safe to assume that the higher a stress-strain curve is, the higher the elastic modulus. Range of strain values were kept at a constant maximum of 0.4, to ensure more accurate comparison.

In Figure 30 and Figure 31, pure titanium has the highest reaching curve, while Ti-5Mo has the lowest. Ti-10Mo and Ti-15Mo also appear to have approximately the same elastic modulus, but at different points of strain.

In terms of elastic limit, both pure titanium and Ti-5Mo exhibit the same level in both test batches, with pure titanium being above the 1,000 MPa mark and Ti-5Mo being below it. As elastic limit is a measure of how much a material can deform reversibly, it is ideal for material to have an elastic limit that stretches much further from greater stress and strain. This thus proves that although Ti-5Mo has demonstrated the lowest elastic modulus, the trade-off is the lesser elastic limit, which will cause irreversible deformation at a lower stress.

The trade-off for a lower elastic modulus is further reinforced by Ti-5Mo possessing the lowest maximum yield point, which is the stress point a material will reach before complete failure. This trade-off however, is not as consequential as elastic limit as the yield point of bone is still much lower than that of Ti-5Mo.

Elastic limit and yield point for Ti-10Mo and Ti15Mo between both batches seem to vary greatly, making it difficult to develop a concise conclusion. The first test batch demonstrates that Ti-10Mo has a lower elastic limit than Ti-15Mo, but a greater yield limit, whereas the second test batch demonstrates the opposite.

The variation in behaviour between Ti-10Mo and Ti-15Mo could be due to the difference in crack propagation throughout the sample. When examining the ductile failure of Ti-10Mo sample from the first batch, sharp edges can be noted on certain corners. This would suggest that the yield point was reached further than it was supposed to, causing a more violent crack propagation that caused an almost torsion-like tear. The comparison of Figure 30 and Figure 31, would support the aforementioned claim, as the first test batch's Ti-10Mo's ultimate yield point is greater than its counterpart in the second test batch.

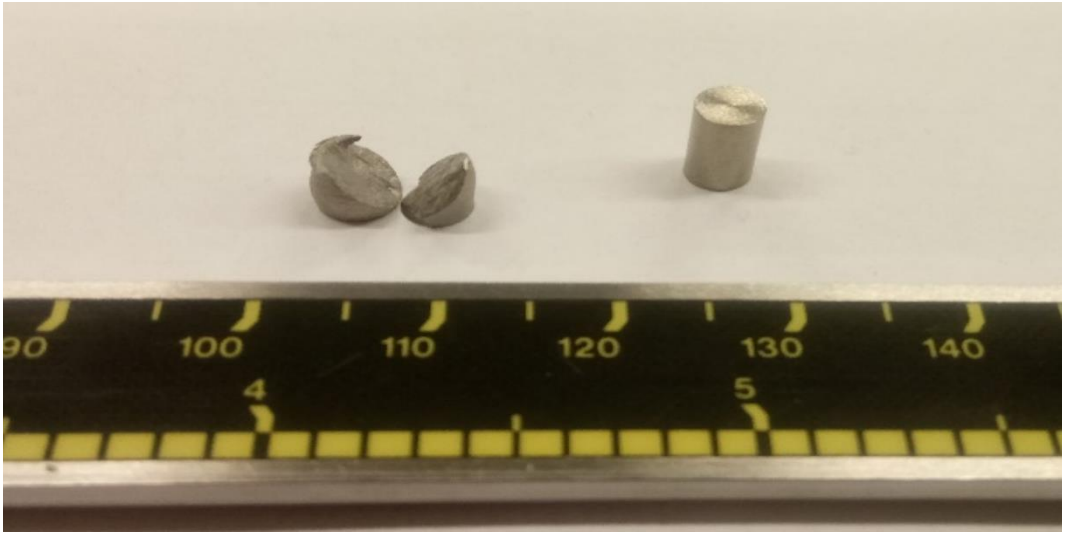

Figure 32: Unusual crack propagation of first test batch's Ti-10Mo (left sample)

It should be noted that although it would appear that the goal of making a titanium alloy possess an elastic modulus of 40 GPa was achieved based on Table 9, this is not the case. The reason such low elastic moduli were achieved with the developed samples was due to their extremely small size, and being placed in a compression tester that often tests samples 3 to 4 times larger. These smaller sizes were unable to distribute the stress uniformly enough throughout its structure, and thus behaved in a manner differently to what would occur had the sample sizes been larger.

5.2.2 Relation to microstructure

Many past literatures claim that in order for a titanium alloy to achieve the lowest possible elastic modulus for biomedical applications, beta-stabilisation must be reached to the highest degree. It is unclear by what measure this is supposed to be, whether it means complete beta-dominance or whether it just implies a beta-presence throughout the majority but not all of the microstructure.

However, upon analysing the results of the current thesis, it would raise the question as to whether beta-stabilisation merely means a presence within the microstructure. As can be seen in both Figure 30 and Figure 31, as well as Table 9, the recurring pattern appears to be elastic modulus drops to the point of 5 wt% molybdenum, but then begins to rise again with increasing molybdenum content.

As was outlined in Section 5.1.2 Ti-5Mo microstructural analysis, Ti-5Mo had only achieved minor phases of developing beta, with alpha phase maintaining a majority of the microstructure, in both equiaxed and acicular form. Relating that back to the compression testing results, it would appear that the state of transitioning between phases achieved the lowest elastic modulus.

In addition, both Ti-10Mo and Ti-15Mo with their transformed beta and beta-dominant states respectively, possessed higher elastic moduli that were similar to that of pure titanium. This implies that once phase change has occurred mostly or completely, elastic modulus no longer decreases but increases instead.

5.2.3 Summary of compression testing

All of the aforementioned would then imply that in order to achieve a low elastic modulus, the alloy must be within the transitional state between alpha and beta phase, or the $\alpha+\beta$ phase, rather than completely reach another phase. This could be attributed to the lattice structure stages mentioned in Section 2.1.2 Common methods, where elastic modulus is directly affected by the morphing of the HCP lattice structure to the BCC lattice structure, most likely due to the lattice structure itself collapsing in order to change.

5.3 Vickers hardness

Analysing Table 10, there is a clear increase in the surface hardness of the titanium alloy as molybdenum content increased. This is achieved through beta grain refinement which was also demonstrated in Section 5.1 Microstructural analysis, where the beta grain boundaries became more characterised as beta-stabilisation increased.

This is proved through the use of the Hall-Petch relationship, displayed in Equation (2).

$$\sigma_y = \sigma_o + \frac{k_y}{\sqrt{d}} \qquad (2)$$

Where:

- σ_y = yield stress of surface (MPa)
- σ_o = the material's resistance to dislocation motion from an applied force (MPa)
- k_y = the material's specific strengthening coefficient (MPa.m$^{1/2}$)
- d = average grain diameter (nm)

As grain boundaries become more refined and uniform in their shape and diameter, the average grain diameter decreases, resulting in an increase in the yield stress that can be tolerated by the surface of the material [5].

Increase in hardness can also be attributed to other factors that occur during the manufacturing process, such as oxygenation during sintering and in the blending and compaction process which causes molecular bonds to strengthen and harden.

5.4 Areas of potential error

As mentioned at the end of Section 5.2.1 Mechanical property interpretation, it cannot be reliably claimed that the ideal goal of reaching an elastic modulus of 40 GPa or lower was achieved due to the size of the samples. Referring back to Equation (1), stress would be far more concentrated than what it would normally be as the surface area undergoing compression force was approximately 5 mm in diameter. Contributing to this, the heights of the samples were less than 10 mm, giving little room for compression strain.

Other forms of potential error stem from more common factors:

- Oxygenation during compaction and sintering, which effects the mechanical properties of the alloy.
- Powder impurities that can cause unusual behaviour in microstructure and potentially effect mechanical properties depending on the size of the impurity.
- Human error with measurement taking, as compression testing machine required input of sample measurements to initiate calculations.

6. Theory from discovery

DISCLAIMER: Upon the writing of this theory, on 19/10/2017, the writer of the thesis was not aware of any theories that could be the same or similar to the theory suggested in the following section. There were no intentions of claiming theories for himself, and if the researchers of said potentially similar theories are offended, the writer of the thesis offers his most sincere apologies.

Contrary to past literature research, the results from the current thesis suggest that larger levels of beta-stabilisation does not guarantee a reduction in elastic modulus. In fact, an argument could be raised as to whether reaching beta-dominance within a titanium alloy increases its elastic modulus to be above that of pure titanium.

Another contributing factor is that the BCC lattice structure does not affect the elastic modulus at all, as a beta-dominant titanium alloy with a BCC lattice structure has proven to possess an elastic modulus on par with pure titanium's HCP lattice structure.

From the two aforementioned points, if a hypothetical graph, where the x-axis is beta-stabilisation and the y-axis is elastic modulus, were to be developed, then the resulting curve would be a positive parabola (where $y = x^2$). The lowest point of the parabola would occur at the point of beta-stabilisation when $\alpha+\beta$ phase is achieved.

This is reinforced by Figure 27, Figure 30 and Figure 31, where the display of Ti-5Mo with its $\alpha+\beta$ phase directly relating to it achieving the lowest elastic modulus in compression testing.

It could then be theorized that in order to achieve a potentially lower elastic modulus, the state at which an alloy is in $\alpha+\beta$ phase must be extended, rather than crossed to reach beta-dominant phase. In order to extend this, the titanium alloy must undergo a process based on alloying metals and manufacturing process that will widen the are $\alpha+\beta$ occupies in the titanium alloy phase diagram, displayed in Figure 1.

7. Recommendations

Due to time and budget constraints, there is much the current thesis was unable to explore in terms of different alloying metals. Referring to Table 4, manganese and iron were the next best candidates for an alloying metal with titanium, and future research would prove interesting with compositions that include one or both of those metals. The development of an alloy composition that extends the α+β phase should also be considered. Incremental increases in the composition of the alloying metal should be varied, potentially at a lower value as an increase in 5 wt% was found to be too great.

Manufacturing method should also become a variable in future research, using processes such as casting or injection moulding. Although those methods are more expensive than standard cold sintering, they also prevent many of the defects caused by sintering such as agglomeration. Each of the other processes used should use varying temperatures to also examine the effects of diffusion on the alloy's molecules.

Finally, post-processing treatment should be utilised in such research, to tailor a titanium alloy even after it has been formed. This can be accomplished through simple methods such as quenching or heat treatment.

Table 11 tabulates all the aforementioned recommendations for better legibility.

Table 11: List of recommendations for future research on titanium alloys

Potential titanium alloys	• Ti-Mo-Fe • Ti-Mo-Mn • Ti-Mn-Fe • Ti-Mn-Fe-Mo
Manufacturing methods	• Electro-magnetic sintering • Injection moulding • Gas atomization • Casting
Incremental increase in alloying metal	• 1 wt% • 2 wt% • 2.5 wt%
Post-processing	• Quenching • Air-cooling • Temperature holding within range of 200-300^{o}C for an hour after sinter completion

8. Conclusion

The purpose of the thesis was to develop a titanium alloy that would be ideal for biomedical application. Although the primary objective of the aforementioned was to reduce the elastic modulus as the main method of increasing biocompatibility, other factors were found to be almost as important in the long-term functioning of a titanium alloy implant.

Through extensive literature research, the most common method of reducing elastic modulus was found to be increasing an alloy's beta-presence, which could only be achieved by alloying titanium with beta-stabilising metals. A range of metals known to be beta-stabilisers were then investigated, noting factors such as whether they actually reduce elastic modulus, increase corrosion- and wear-resistance, and maintain no toxicity during the life of the implant within its recipient.

Once sufficient research on different metals was completed, a criteria and decision matrix were developed to choose the most suitable metal to alloy with titanium. The resulting decision was molybdenum.

In order to see more immediate and noticeable changes with varying compositions, the increase in molybdenum content was to be in increments of 5 wt%, resulting in the compositions being Ti-5Mo, Ti-10Mo and Ti-15Mo, as well as pure titanium for reference in result comparison. The alloys would then be developed through use of PM sintering, with two sample batches for compression testing, one sample batch for microstructural analysis, and one sample batch for Vickers hardness testing.

Through microstructural analysis, it was proven that molybdenum is a strong beta-stabiliser that was able to change titanium's natural alpha phase into beta phase, with retained acicular alpha and refined beta grain boundaries.

Vickers hardness was found to have increased with increasing molybdenum content mainly due to grain refinement outlined in the microstructural analysis. Other reasons to contribute towards increased surface hardness could also be due to the oxygenation that occurs during manufacturing processes.

The most noteworthy discovery however, occurred during the analysis of compression testing results. Even though beta-presence increased with increasing molybdenum content, elastic modulus did not decrease as much as past literature would suggest. Instead, the lowest elastic modulus achieved was at Ti-5Mo, where beta-presence was still developing within the microstructure, and beta grain boundaries were unrefined. After that, elastic modulus began to increase to the same point as pure titanium.

The combined findings from the microstructural analysis and compression testing would lead to the conclusion that beta-stabilisation does in fact, decrease the elastic modulus, but only to a certain extent. This extent is defined within the stage of phase transition between alpha to beta, where the microstructure is termed as $\alpha+\beta$ phase because of its shared composition of alpha and beta sections.

Vickers hardness testing proved to increase the surface hardness of the titanium alloy, which results an increase in wear-resistance. Corrosion-resistance was unable to be examined as the means for procedurally testing it were out-of-scope, however past literature outlined that molybdenum does impart a passive protective-film, and thus meets the requirement of being corrosion-resistant.

The resulting alloy of Ti-Mo from the current thesis was able to achieve its goals of lowering elastic modulus, and increasing corrosion- and wear-resistance, with elastic modulus being of lesser extent. This thus proves that beta-stabilising metals do in fact, change titanium alloys to be more appropriate for biomedical application.

9. References

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10. Appendix

Appendix A: Images of microstructural analysis

A1 – x20 magnification

Pure Ti:

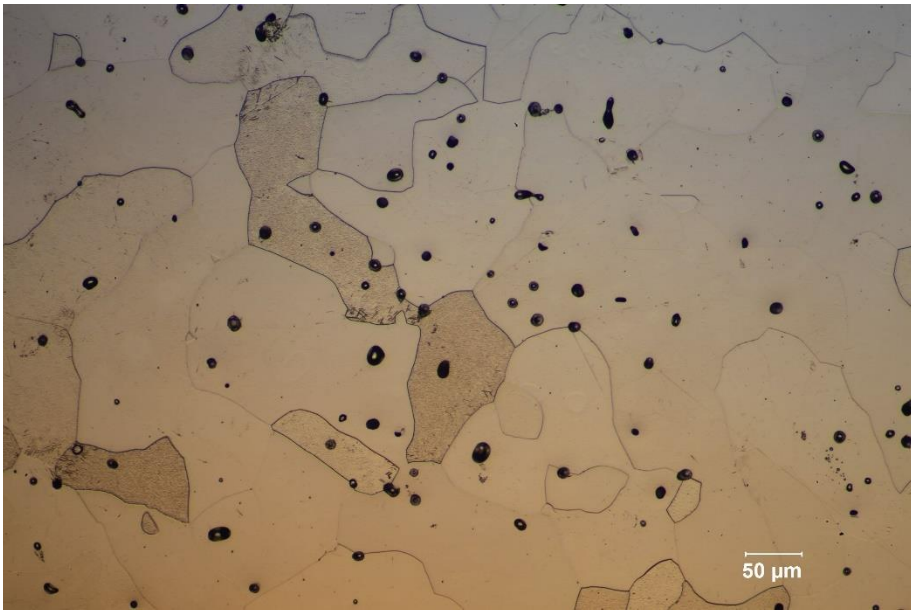

Figure 33: Pure Ti microscopic image at 20x magnification, first image

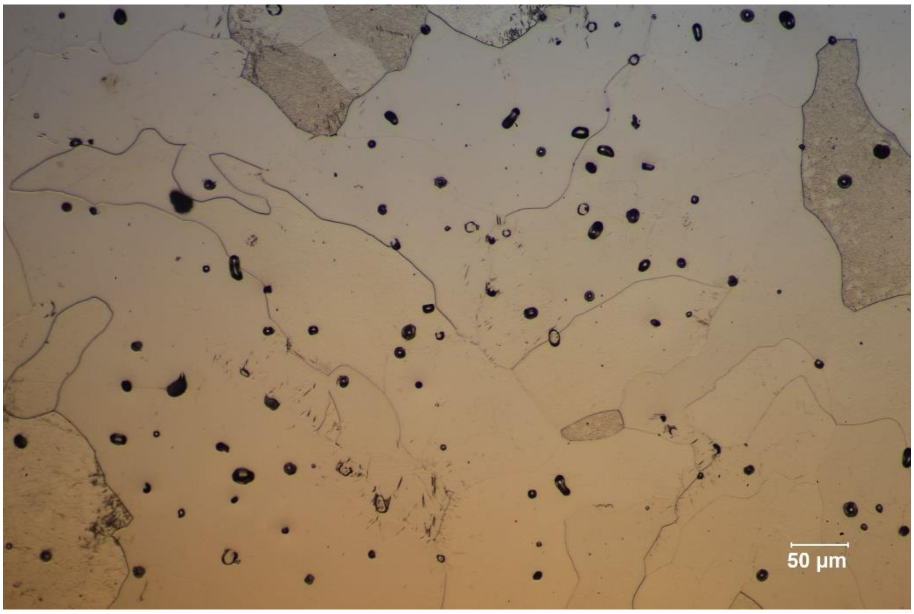

Figure 34: Pure Ti microscopic image at 20x magnification, third image

Ti-5Mo:

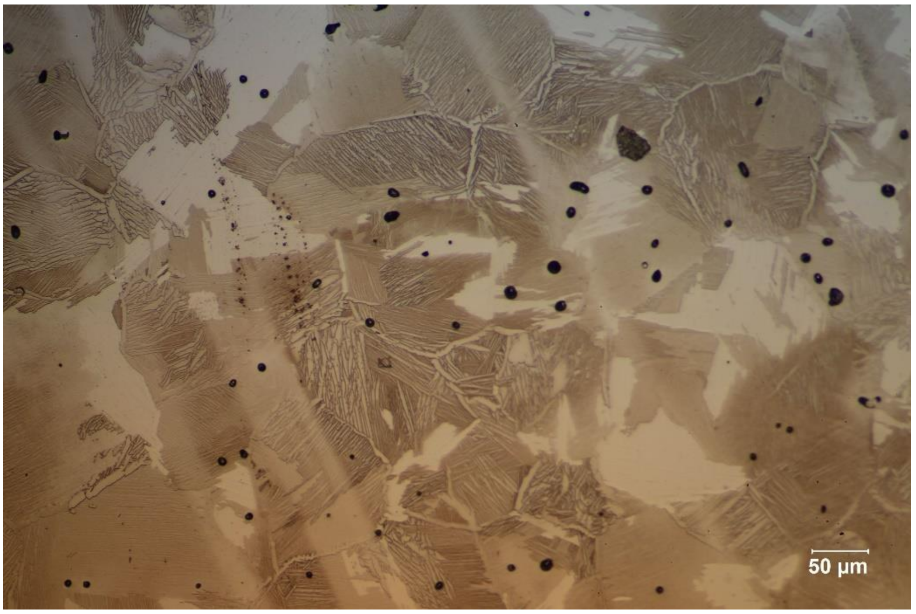

Figure 35: Ti-5Mo microscopic image at 20x magnification, second image

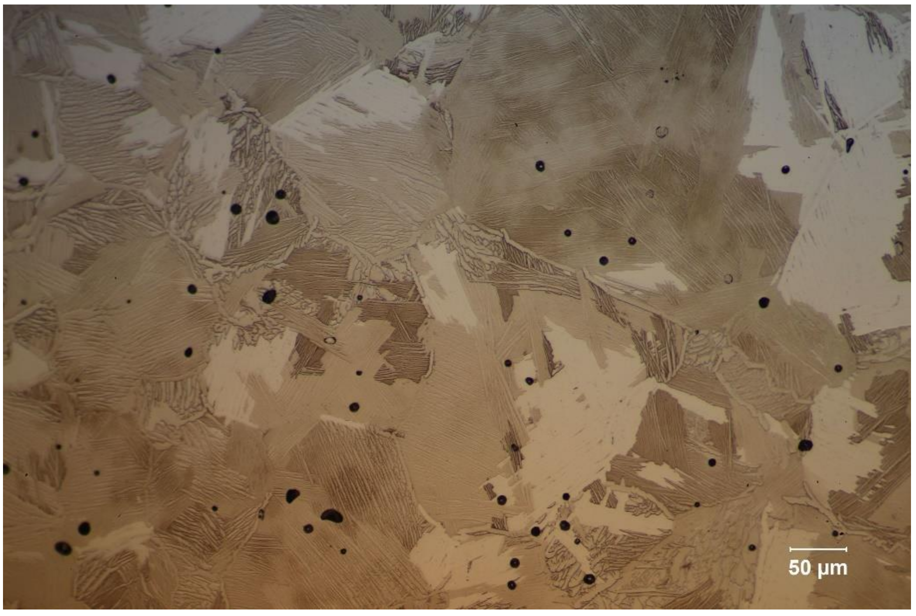

Figure 36: Ti-5Mo microscopic image at 20x magnification, third image

Ti-10Mo:

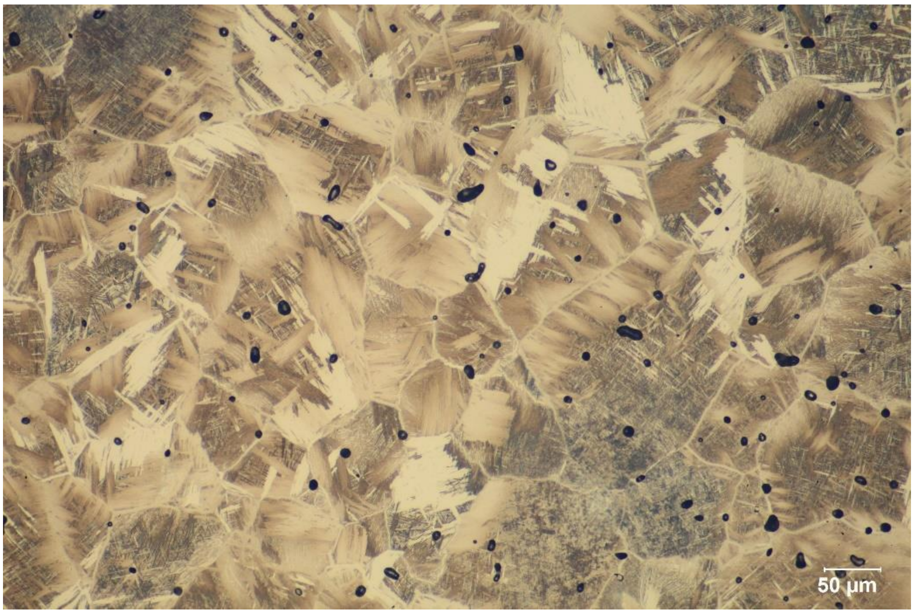

Figure 37: Ti-10Mo microscopic image at 20x magnification, first image

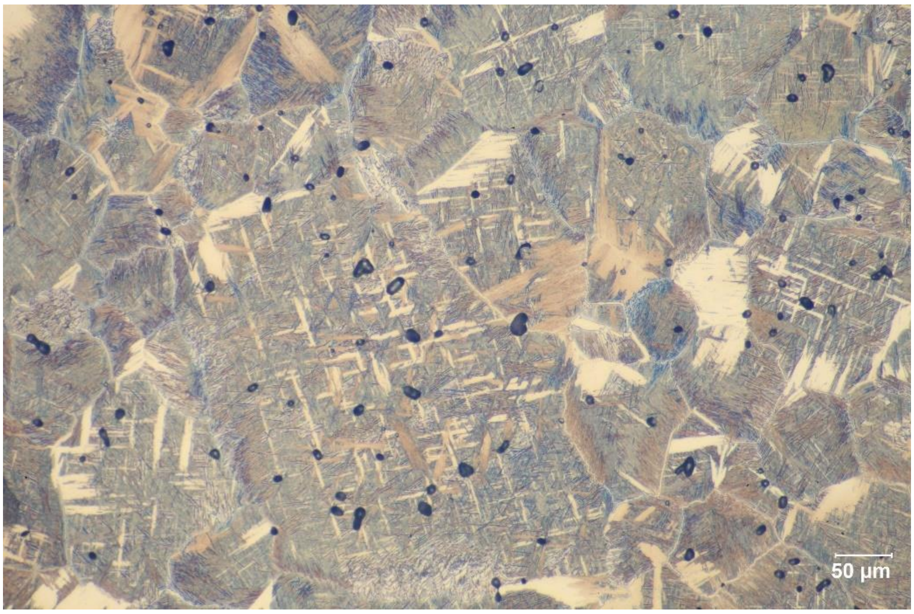

Figure 38: Ti-10Mo microscopic image at 20x magnification, third image

Ti-15Mo:

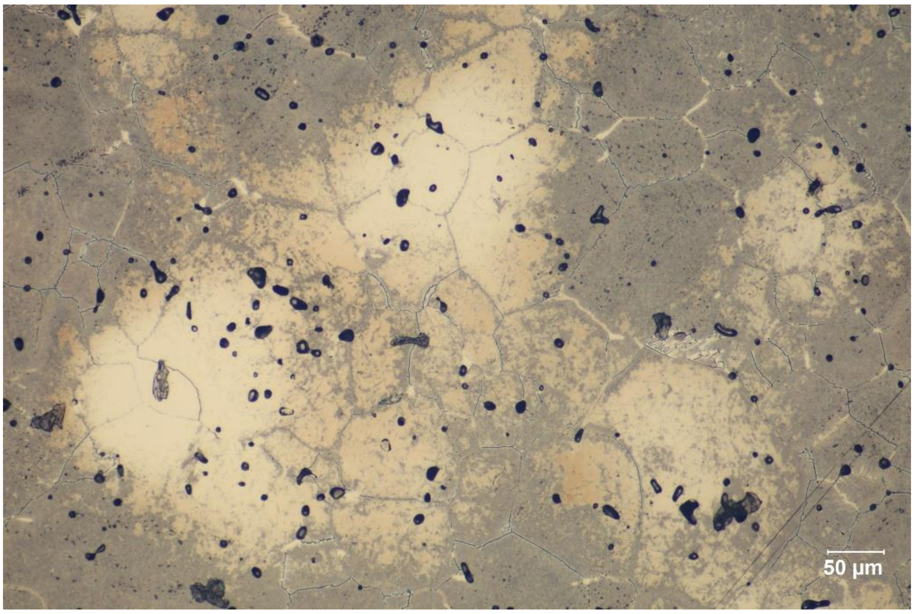

Figure 39: Ti-15Mo microscopic image at 20x magnification, first image

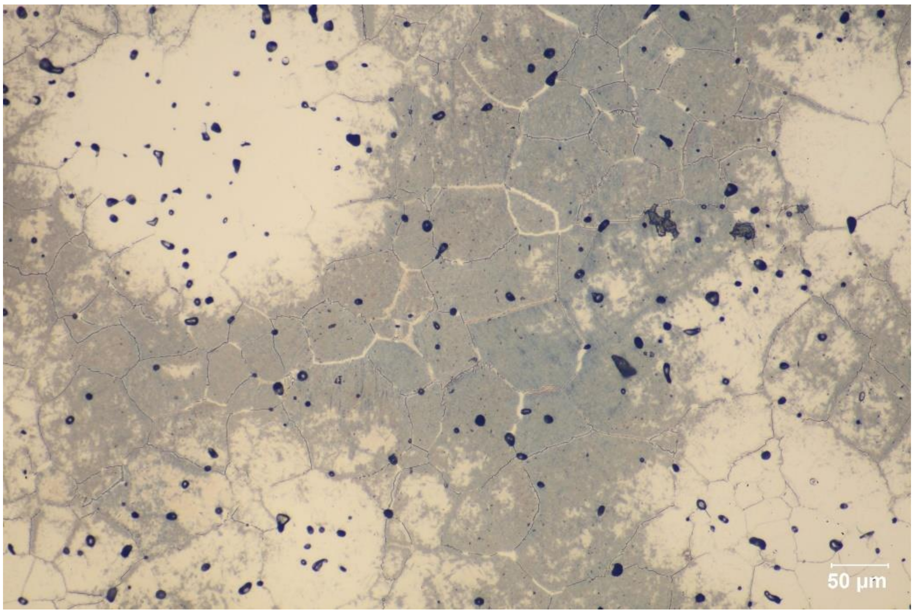

Figure 40: Ti-15Mo microscopic image at 20x magnification, second image

A2 – x50 magnification

Pure Ti:

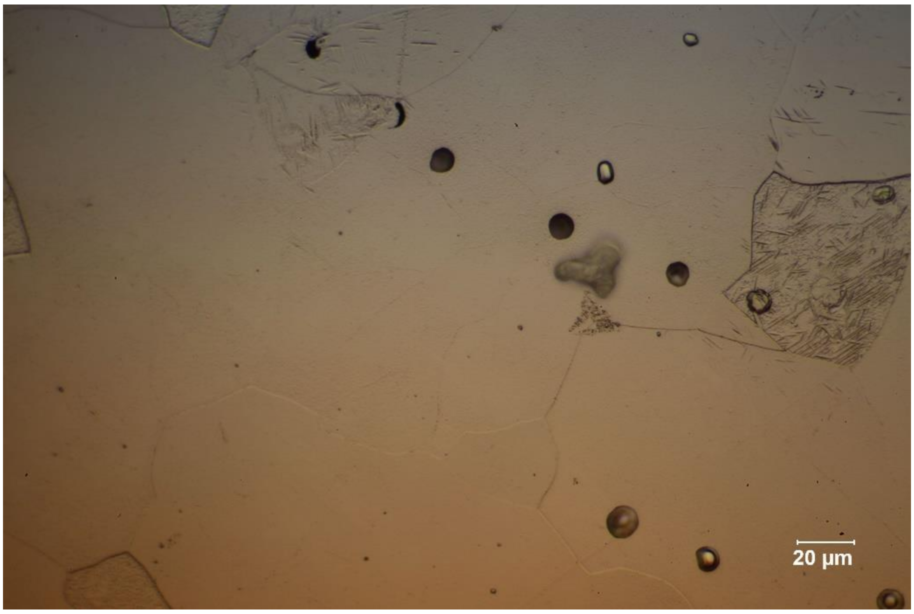

Figure 41: Pure Ti microscopic image at 50x magnification, second image

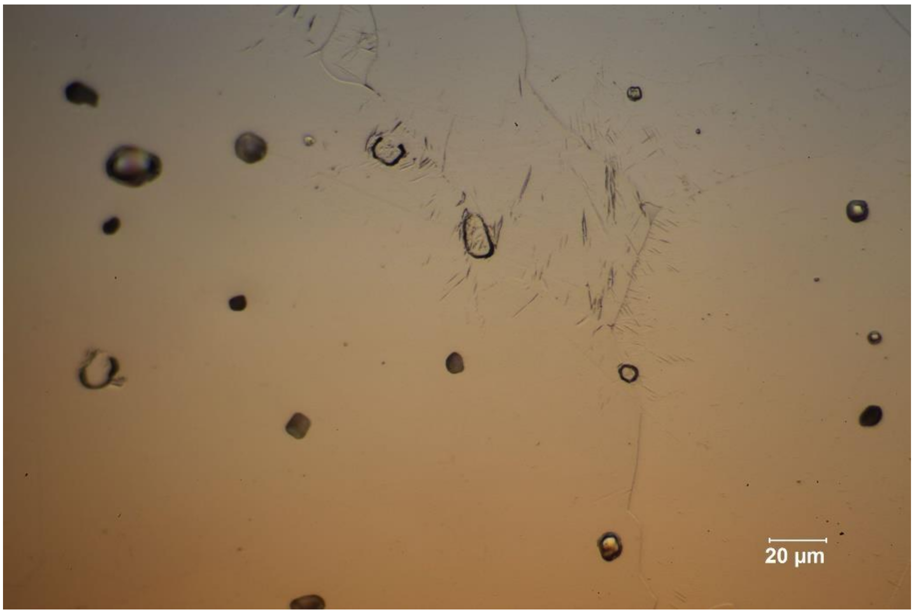

Figure 42: Pure Ti microscopic image at 50x magnification, third image

Ti-5Mo:

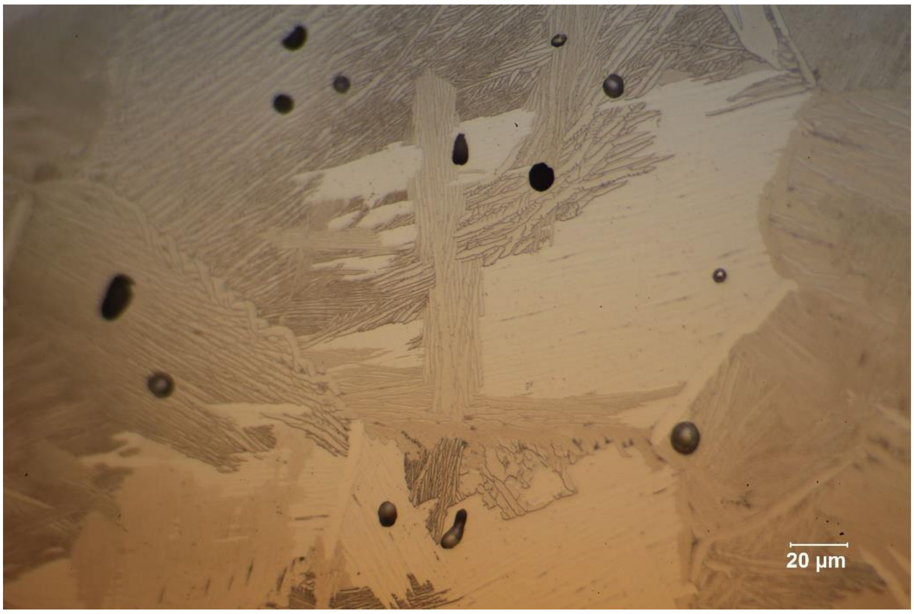

Figure 43: Ti-5Mo microscopic image at 50x magnification, first image

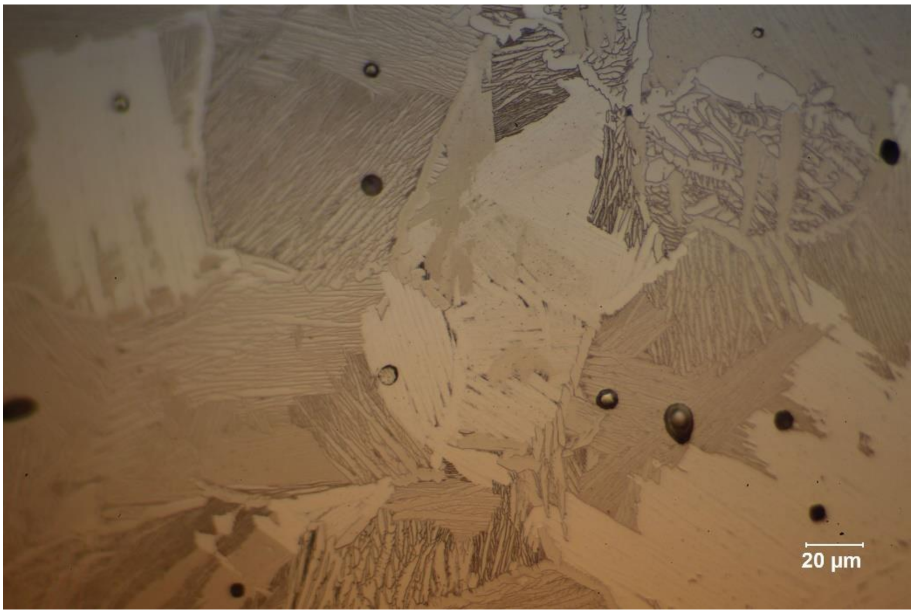

Figure 44: Ti-5Mo microscopic image at 50x magnification, third image

Ti-10Mo:

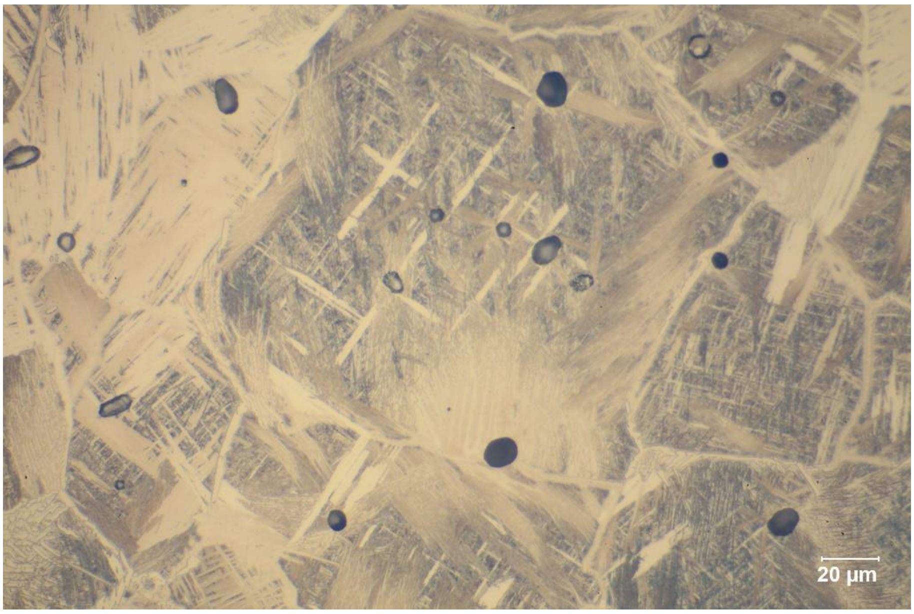

Figure 45: Ti-10Mo microscopic image at 50x magnification, first image

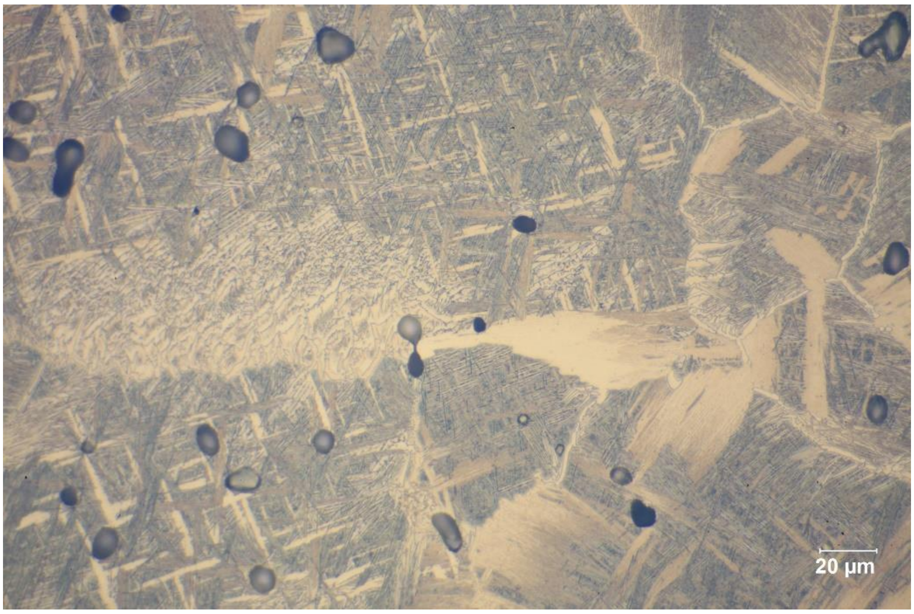

Figure 46: Ti-10Mo microscopic image at 50x magnification, third image

Ti-15Mo:

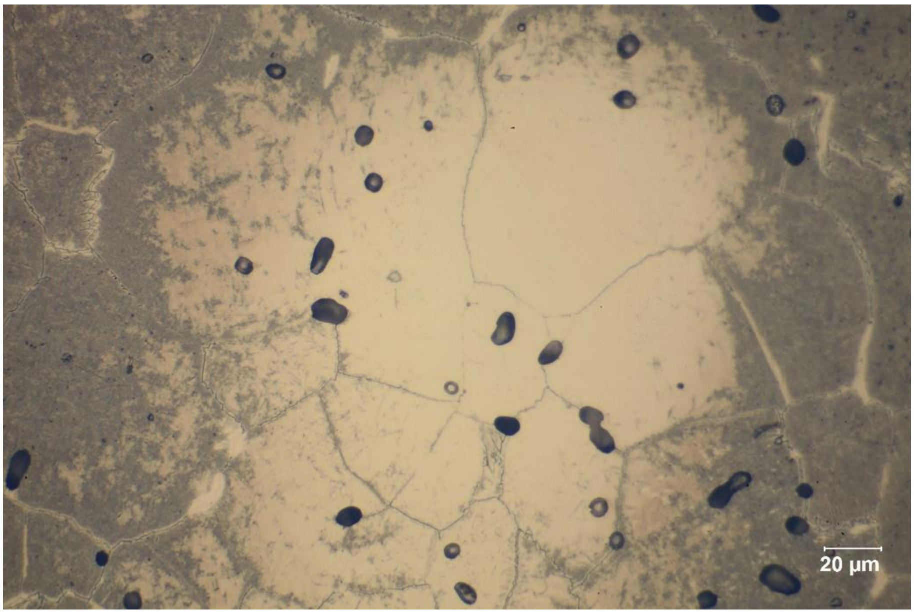

Figure 47: Ti-15Mo microscopic image at 50x magnification, first image

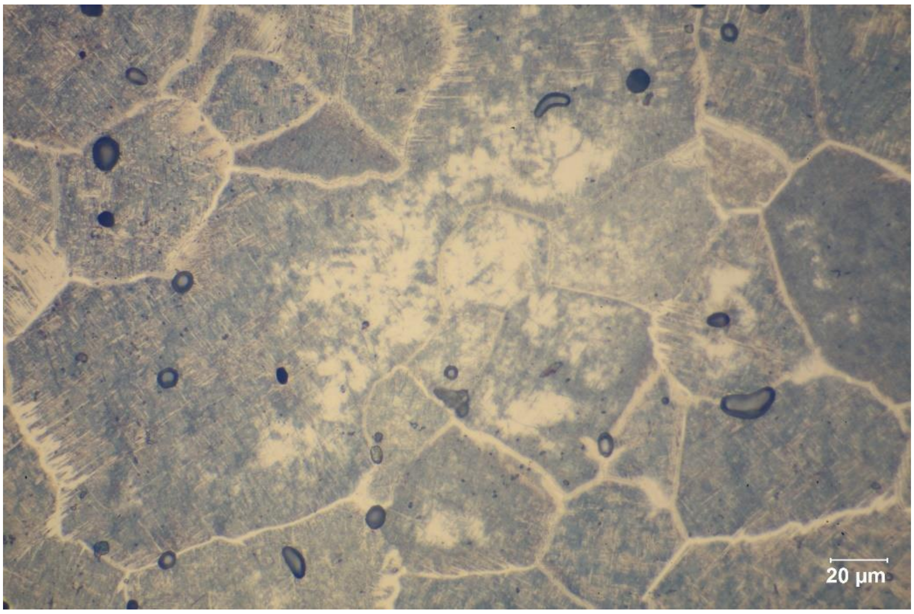

Figure 48: Ti-15Mo microscopic image at 50x magnification, second image

Appendix B: Vickers hardness test results

B1 – Pure Ti and Ti-5Mo

Pure Ti						Ti-5Mo				
	Reading 1	Reading 2	Reading 3	Average			Reading 1	Reading 2	Reading 3	Average
Trial 1	255	255	255	255		**Trial 1**	260	259	258	259
Trial 2	290	288	289	289		**Trial 2**	309	320	316	315
Trial 3	263	261	262	262		**Trial 3**	267	267	267	267
Trial 4	320	312	321	317.6667		**Trial 4**	292	292	292	292
Trial 5	326	330	332	329.3333		**Trial 5**	322	327	325	324.6667
Trial 6	283	272	272	275.6667		**Trial 6**	284	295	284	287.6667
Trial 7	321	326	320	322.3333		**Trial 7**	339	347	346	344
Trial 8	268	273	271	270.6667		**Trial 8**	313	297	302	304
Trial 9	315	315	315	315		**Trial 9**	301	303	307	303.6667
Trial 10	203	202	200	201.6667		**Trial 10**	279	275	280	278
Trial 11	325	333	316	324.6667		**Trial 11**	319	320	326	321.6667
Trial 12	286	284	285	285		**Trial 12**	312	308	304	308
Trial 13	298	298	298	298		**Trial 13**	320	327	321	322.6667
Trial 14	286	291	291	289.3333		**Trial 14**	277	286	283	282
Trial 15	285	291	288	288		**Trial 15**	310	315	318	314.3333
		Total sample avg		288.2222				Total samples avg		301.5778

Figure 49: Vickers hardness results for Pure Ti and Ti-5Mo

B2 – Ti-10Mo and Ti-15Mo

Ti-10Mo						Ti-15Mo				
	Reading 1	Reading 2	Reading 3	Average			Reading 1	Reading 2	Reading 3	Average
Trial 1	330	327	336	331		**Trial 1**	372	375	370	372.3333
Trial 2	365	361	364	363.3333		**Trial 2**	365	363	353	360.3333
Trial 3	325	324	326	325		**Trial 3**	383	384	387	384.6667
Trial 4	334	342	339	338.3333		**Trial 4**	399	396	387	394
Trial 5	314	317	316	315.6667		**Trial 5**	374	375	376	375
Trial 6	343	342	341	342		**Trial 6**	359	362	365	362
Trial 7	345	335	337	339		**Trial 7**	433	415	426	424.6667
Trial 8	355	366	361	360.6667		**Trial 8**	365	360	350	358.3333
Trial 9	295	307	296	299.3333		**Trial 9**	422	431	421	424.6667
Trial 10	348	348	348	348		**Trial 10**	391	393	390	391.3333
Trial 11	306	310	305	307		**Trial 11**	402	398	400	400
Trial 12	334	333	331	332.6667		**Trial 12**	407	407	407	407
Trial 13	332	330	337	333		**Trial 13**	402	406	399	402.3333
Trial 14	368	362	363	364.3333		**Trial 14**	409	408	404	407
Trial 15	338	340	338	338.6667		**Trial 15**	416	406	409	410.3333
		Total sample avg		335.8667				Total sample avg		391.6

Figure 50: : Vickers hardness results for Ti-10Mo and Ti-15Mo